Indian Institute of Technology
Kanpur

NP-TEL
National Programme
On
Technology Enhance Learning

Course Title
Advanced Characterization Techniques

Lecture-01

By…
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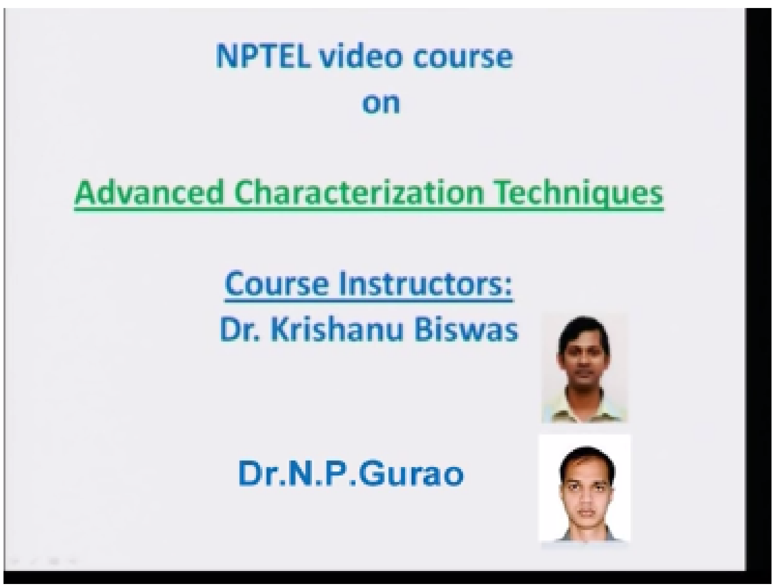

Hello welcome to the NPTEL video course on advanced characterization techniques myself Dr. Krishanu Biswas this course will be designed by myself and one of my colleagues in the Department of material science engineering at IIT Kanpur, so at least 20 lectures of this course will be delivered by me and the remaining 20 will be delivered so you can see that in the next slide the outline of the course.

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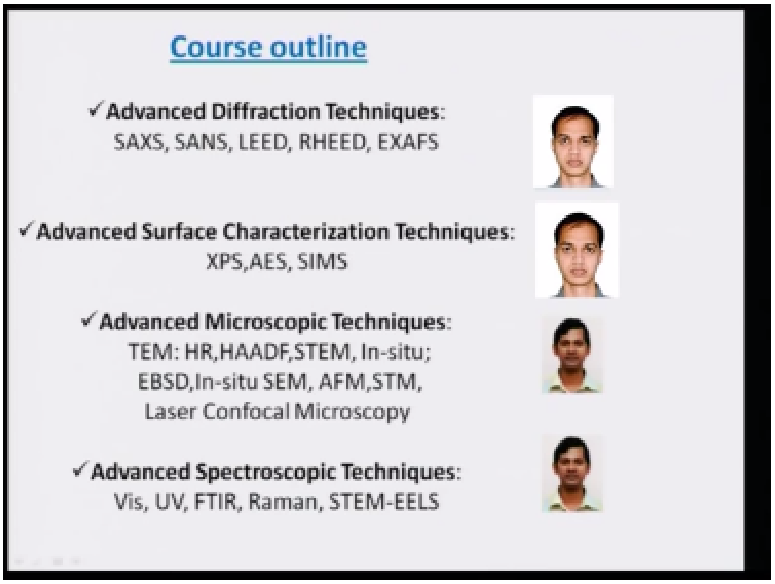

Which is given this course contains different advanced characterization techniques first one advanced diffraction techniques includes small angle extracting fractions small angle neutron scattering low-energy electron diffraction read and as well as accepts second one which is again advanced surface characterization techniques exclusively for XA fertilization spectroscopy Osier photoelectron spectroscopy and secondary iron mass spectroscopy so therefore II students will consist of 20 lectures second mostly microscopic techniques.

And the spectroscopic techniques so when the microscopic techniques we are going to discuss in detail about transmission electron microscopes including high resolution hiring an area dark field imaging steam institute microscopic techniques like in situ SEM and institute TM electron back scatter diffraction FM that is atomic force microscopy STM that is scanning tunneling microscopy as well as laser Confocal microscopy this will be delivered by me and I am going to start the first lecture of these in the next one and the last part.

Of the course is basically on advanced spectroscopic techniques which are very important nowadays for research on nano materials and many other things this includes visible ultraviolet Fourier transform FTIR Raman spectroscopy as well as in the microscopic techniques the same electron energy loss spectroscopy so therefore this course basically talks about the advanced calculation techniques, so to understand this course one needs to know the basics of the normal characterization techniques that is why the calculations course requirement for these kind of course must be the basic knowledge.

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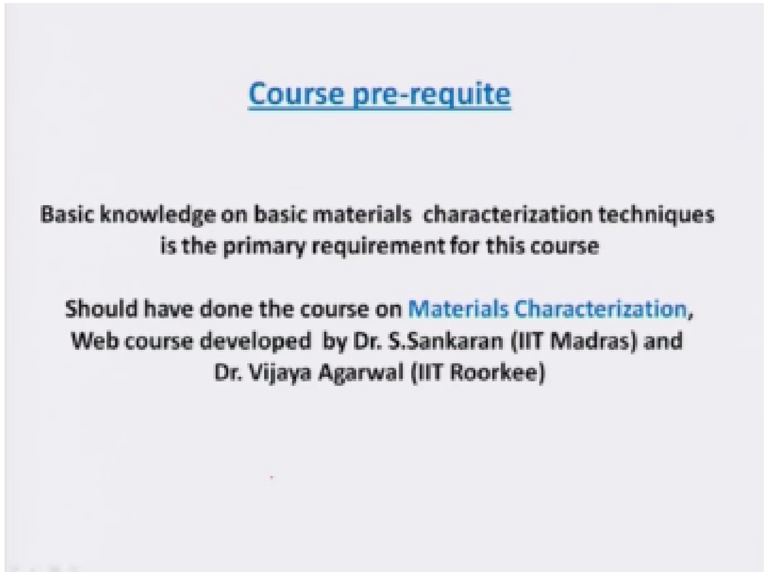

On the basic material characterization techniques and this kind of course is already been developed by Dr. S. Sankaran from IIT Madras and Dr. Vijaya Agarwal of IIT Roorkee as a wave course as a part of the NPTEL lecture series so whatever our discussions. I am going to do for the different characters in techniques has lot of inputs from this metal characterization course by two of our colleagues so now we shall discuss the detail lecture wise breakup of this course this is basically required to tell you that how the different lecture modules will be developed.

So first lecture which is today. I am going to talk about relevance of advanced characterization techniques for materials development.

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Topic	No. of lectures
Introduction to the course: Relevance of advanced characterization to materials development, scientific understanding of phenomena in materials technology	1
Advanced Diffraction Techniques:	
Introduction; X-Ray, their production &properties	3
Review of basic diffraction theory;	
Various SAXS techniques and its applications in characterizing material	4
SAXS	
GISAXS	
LEED and RHEED	
EXAFS, SEXAFS/NEXAFS	2
Properties of neutron radiation; neutron sources;	
Small angle neutron scattering; Examples	
Advanced Surface Characterization Techniques: XPS, AES and SIMS	
Importance of surface characterization techniques	3
Physical principles of XPS, Photoelectric effects;	
Instrumentation, XPS patterns; Spin orbital Splitting;	
Quantitative analysis, Chemical effect, Chemical shift, XPS imaging	
Auger electron generation; Principle, Chemical effect, Quantitative analysis, Depth profiling, Applications	3
Static and Dynamic Secondary Ion Mass, Common modes of analysis, Depth Profiling, quantitative and Qualitative analysis ,Comparison surface analysis techniques	1
Advanced Microscopic Techniques:	
Introduction; Electron-materials interactions;	1
TEM: HR,HAADF,STEM,In-situ TEM;	4
SEM, EBSD,In-situ SEM	2
AFM, STM,	2
Laser Confocal Microscopy	1
Advanced Spectroscopic Techniques:	
Introduction; Electromagnetic spectroscopy; UV-Visible Spectroscopy; Photo-luminescence spectroscopy; Infra-red spectroscopy;	7
Raman; STEM; EELS	3

Basically these are required for scientific understanding of the different phenomena in the material science engineering then the next two parts as advanced diffraction techniques and advanced application techniques the detailed lecture wise wakeup is shown in the slides and this will be discussed by Dr. Gautama in different lectures, so I will straight away go to the last two topics in advanced microscopic techniques first lecture of that will be devoted on electron material interaction this will be followed by four lectures.

On transmission electron microscopy mostly on high resolution and other allied techniques remaining two lectures will be delivered on SEM related things like in situ SEM and the electron back scatter diffraction whereas atomic force microscopy and scanning transmission canning colony microscopy will be dealt in two lectures and lastly the laser Confocal microscopy will be discussed in the advanced spectroscopic techniques seven lectures will be delivered on electromagnetic spectra scope is mostly EB visible.

And photo luminescent spectroscopy infrared spectroscopy and also the Raman spectroscopy last lectures of this course last three lectures of this course will be delivered on stands and scanning transmission electron microscopy related spectroscopic techniques that ills and ideas that will sum up twenty lectures in these two techniques well as any other course we need to have some reference and the books so in these slides.

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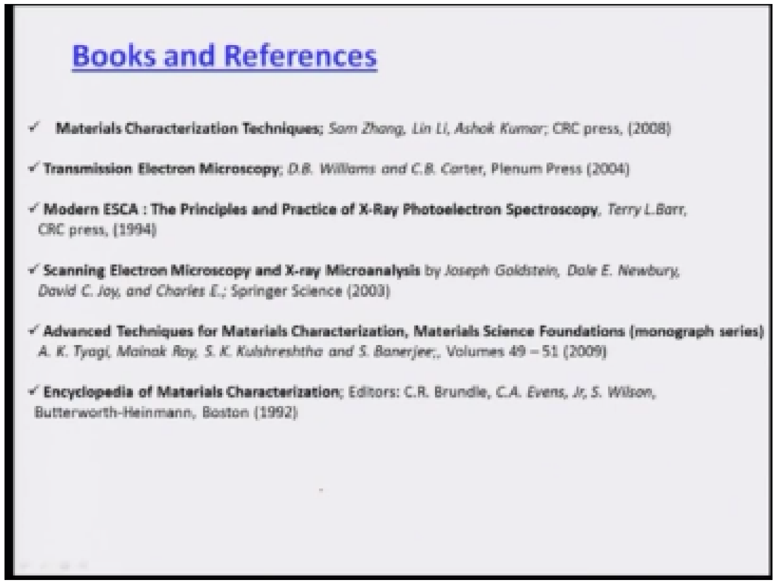

I am showing you some of the important books these are not exhaustive there are many other books available in the libraries or even the market but. I am listing down the most important ones and the ones which I am going to use and Dr. Gautama also going to use is going to use Forrest lectures the first one is the material characteristic techniques by Zhang Li and come on it is published in 2008 which we will talk about all the characters and techniques in brief second one is basically on tactic electron microscopy by Williams and Carter.

Third one is on modern Eska the principles and the practice of excess photoelectron spectroscopy by per from again CRC pace fourth one is on scanning electron microscopy and x-ray microanalysis by Goldstein and others fifth one is an advanced techniques for metal characterization it is basically a monograph by the material science foundation edited by A.k. Tyogi, Mainak Roy ,S.K.Kulshrehtha and S. Banerjee, the all the techniques can be easily obtained our rather can be easily accessed in encyclopedia called encyclopedia.

Of material characterization edited by Brundle events Wilson published by butterworth-heinemann Boston and this is a very exhaustive book so therefore. I will not prefer you at the beginning to go into the encyclopedia rather than concentrate on the different books on different subject matters obviously first lecture is on relevance of the character Asian techniques.

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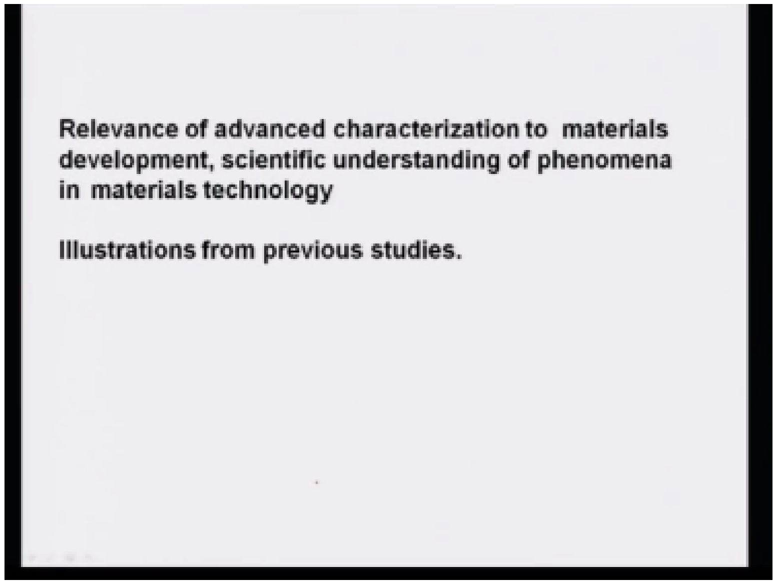

So giving you an idea or the core structure and the modules, I have to now discuss why one needs to study such a kind of course those of you who have some idea about the characterization must have seen different characterization labs in the different parts of our country coming up as a part of schemes by Department of Science and Technology and many cases even by other allied organizations these centers are actually called characterization centers there are centers in many IITs even in IIT Kanpur IIT Madras in earnest of Science Bangalore NCL Punai.

And many other places where in one roof all the different characters and techniques are housed so therefore you have those of you have visited these places have got some idea about the different sophisticated characterizing tools are used for calculation of materials so I am not going to discuss the way the characterization techniques can be used rather. I will give you some examples from our own study where characterizations were used by our own students to understand the material sands phenomena.

In different processes and also to process different kinds of material these are all done by our students so therefore these are all taken from my own research group and most of these works are done on very small sized particles like nano particles or nano materials, so I will one by one I will illustrate to you that how important it is to use characterization techniques before that I would like to tell you then the last ten years there is a huge improvement in the characterization techniques as far as the limits of the use of this instrument are concerned.

One of the example is the high-resolution transmission electron microscope in late 90s even at the beginning of the 21st century the resolution limits for Tames electron microscopy used to be in a Armstrong level that is above 1 Armstrong ,but because of the advent of new technologies especially the aberration corrected microscopic technologies we could have very high resolution we can reach resolution of 1 6 Armstrong so this barrier are breaking the resolution level below 1 Armstrong was possible.

Only because of the technological advance and I will show you in some of my lectures how this microscopes can be used to decipher different structures and also to understand the problems of material science so let me give you the examples the first example is on.

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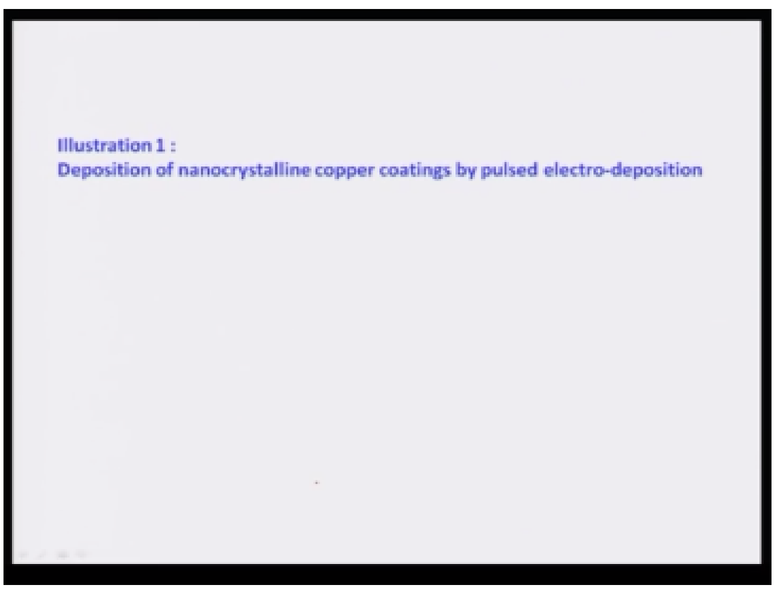

Depletion of nanocrystalline copper coatings by pulsed electro deposition technique we know that nanocrystalline copper is very important because nowadays in the electronic industry the connectors are normally made by copper previously those connections and the visas used to be made from gold or aluminum or tungsten but technology has seen a rapid change because copper can be easily deposited in those positions of the semi-conductor devices where the connections are required.

So basic requirement for those kind of connections are very strong and very good electrical conductivity of copper not only that the brightness of the deposits by the electrode depletion should be also very good these requirements can only be satisfied if one develops a nano crystalline copper deposits by using a pestle technique called pulse electro deposition this is as shown in the slides.

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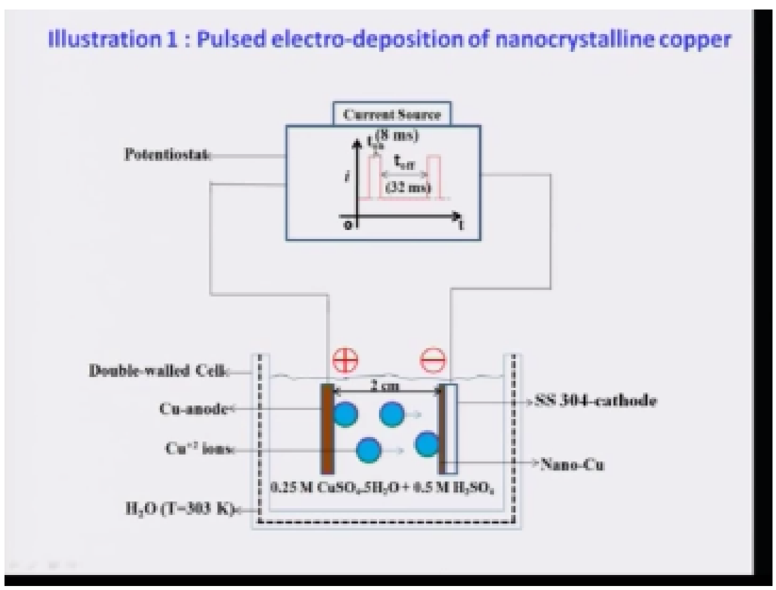

What you can see is a first one is basically showing the pulse electro deposition technique where current is plotted as a function of time in this case we use pulsed current for about eight millisecond followed by no current supply for about 30min seconds that means the T on the current is on for all the eight millisecond followed by T off of 32 milliseconds then again the current is on for about 80 milliseconds eight milliseconds so therefore by using this pulsed current cycles one can deposit this kind of nano crystalline copper.

I am not be able to go into detail of this diffusion technique except to show you that this can be done in a normal electrochemical cell double wall cell where anode is a copper and cathode is basically stainless steel c04 grid so therefore if we apply current when this hole the gold both electrodes are dipped into a copper sulfate sulfuric acid solution at about 303 degree Kelvin one can see the depositions of copper from anode to the cathode taking place and by that one can deposit different copper of different thickness.

On the stainless steel substrate the reason for using stainless steel is that it is easy to peel up the copper flame from the stainless steel substrate to give an example.

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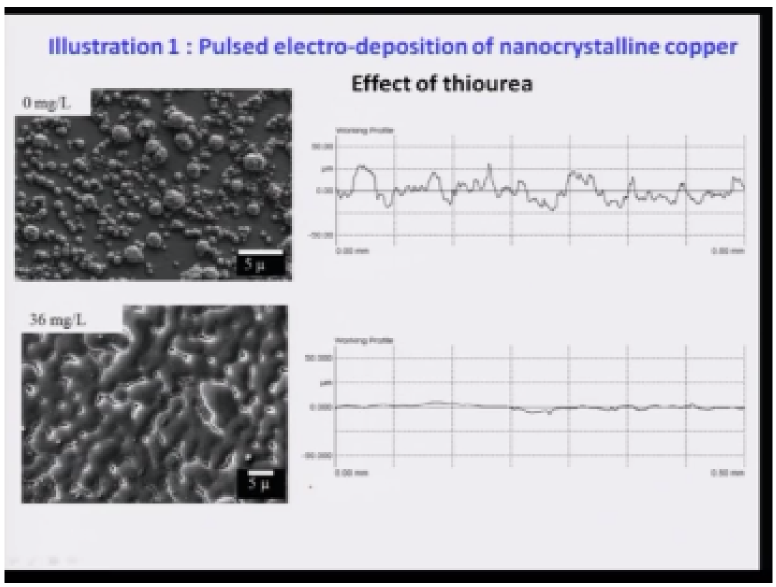

How this deposition is done is shown in this case or normally if we deposit copper using pulse retribution technique we do not get nano crystalline grain sizes so therefore many cases we add different additives and this additives can be anything like PG polyether I call or any other sulfur compounds like tayo urea thyrsus most notably used because it is nothing but a sulfur-containing compounds and these sulfur-containing compounds can change the deposition kinetics so and so that it is possible to develop an anarchist in copper grains in the deposit.

The reason for this was not known people have seen this happened but why and how the tyro is affecting the deposition process so the nanocrystalline copper gain can be obtained was not known so one of our students in my research group started looking at this problem and found several interesting observations to understand the effect of Tyria on the deposition of nano crystalline copper on 304 stainless steels first I will show you two SEM pictures scanning electron microscopy images of the thin flame deposited on stainless steel substrate.

The first one is deposited when there is no thiourea so you can see very discontinuous deposits and obviously using laser scanning profilometer one can measure the roughness to characterize quantitatively how rough the deposit which is shown on the right side of the picture you can clearly see that roughness is very high some cases mean roughness can be augur of several tens of micron so therefore if you know that heavier deposits are very discontinuous and also rough or not and if you add very small amount of thiourea about thirty-six milligram per liter deposits become very smooth one can see.

In the picture here deposit is very smooth and also if we measure the surface roughness is very high sorry very low but that means roughness can be easily controlled by the theory addition and this is where required when the copper is deposited on any kind of PCB board our visas are in the electronic circuitry so this was observed very nicely by using a scanning electron microscopy technique followed by laser surface for a limiter these two are the advanced characterization techniques used in many cases the common idea of depletion model.

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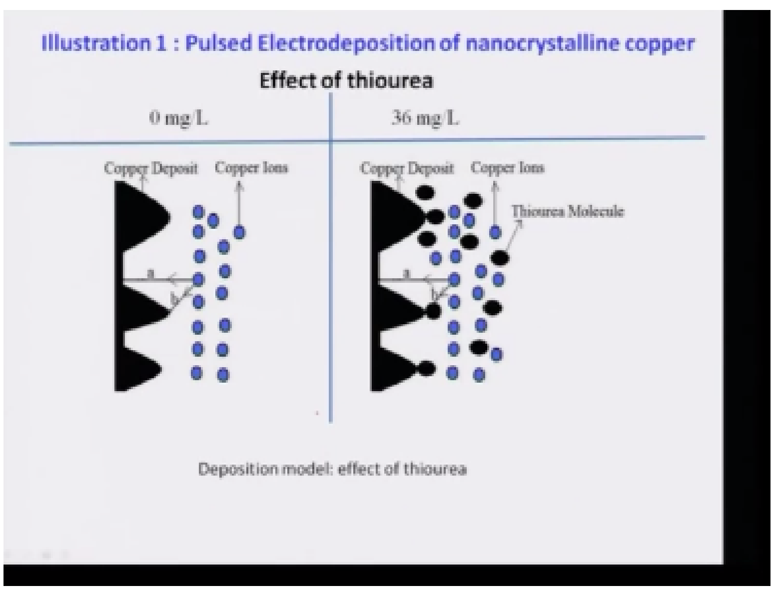

Is this will help us understanding how thiourea actually affect the diffusion process so when there' is no thiourea obviously substrate will have lot of surface steps on this like any other surface and that the copper ions will move to the different places of the substrate most notably the copper annually will to move on the high or on the crest of the surface instead of Tufts here so thereby depositing on the crest and making them thicker and thicker and creating deposits the one which I have shown you which is kind of discontinuous on the surface.

If I add heavier molecules into the deposit or into the electrolyte rather the thiourea molecules will basically gets deposited on the surfaces of on the crest and that is how they will not allow the copper irons to go on this earth on this crest instead of daily force to go on the Tufts here and because of that the stops will get filled up and you will form a very continuous deposit so that roughness can be reduced very easily this is the common idea which is been reported in the literature but knowing this does it happen in the actual sense.

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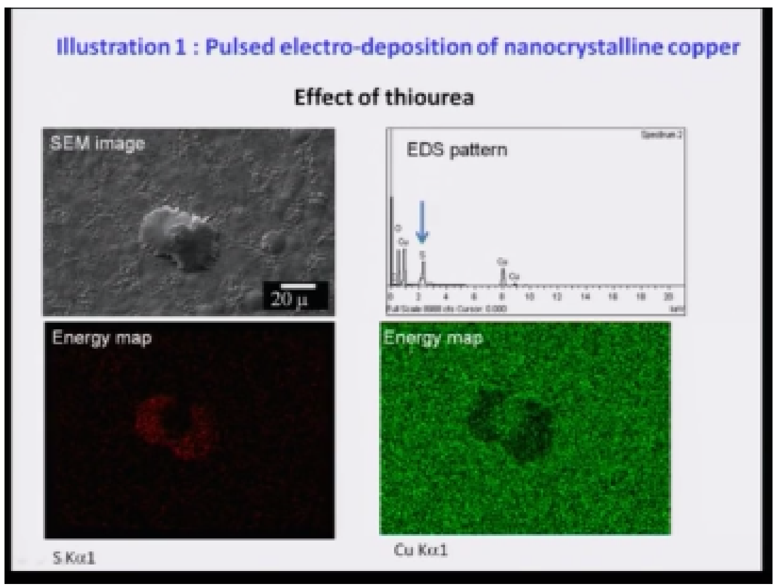

To do that we have done very careful spectroscopic analysis using a scanning electron microscope the picture here shows the scanning electron microscopy image of the copper deposit in which there are too large and also very you know something which is protruding out of this of the surface of the deposit if we do EDS analysis that is the energy dispersive spectroscopy analysis attached to the SEM which is nothing but a spectroscopic technique using electron beam one can see the presence of sulfur peak very easily coming from the surface.

Not only that one can even further analyze this using the index mapping if you do a text mapping on the whole surface you see sulfur is basically segregated on the surface remember the sulfur is a part of the trireme molecule and this theory of molecule is formula is given by NH 2 whole to CS so therefore sulfur is obviously it is part of the molecules wherever you see sulfur we can assume that every is present there because copper does not contain any sulfur so that is why you can say that the model.

Which has been developed by different scientists can be proved by different kinds of spectroscopic technique if you see in the copper Mac it will uniformly distributed on the whole surface except the position where there is a protrusion which is spin can be mapped very easily using the scanning electron microscope image.

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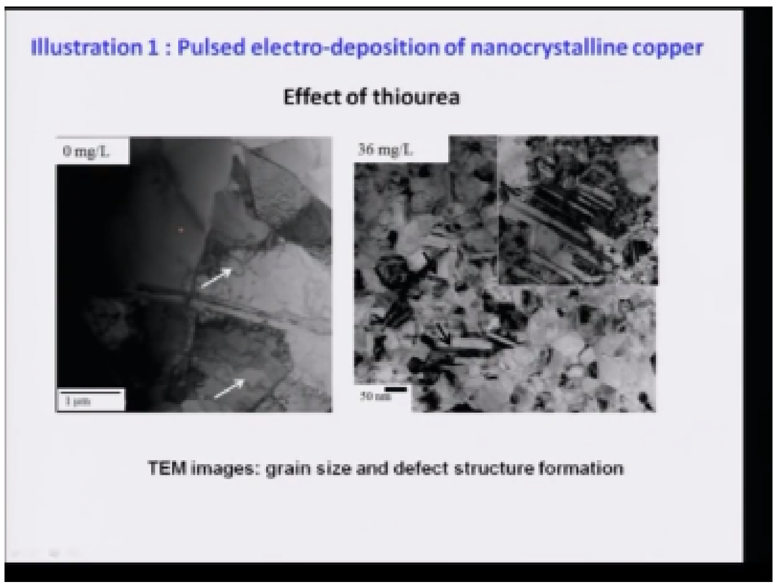

Not only that one can go on use of different other techniques this one is called transmission electron microscopic technique which can be used to know the fine-scale structure inside these deposits the left one is showing you the electron microscopic image in the rightful mode of the sample deposited without additional penny thiourea where you can see that very large grain this bar is a microns bar one micron so grains are very large of the order of 2 to 3 micron and they contain a lot of dislocation inside.

It which are marked by this white arrows on the other hand if we are higher up 36 milligram per liter we can see the greens is extensively reduced from micron size to nanometer size if this much length is 50 nanometer so most of our grains are less than hundred nanometer size so this really tells us theory affects the grain size reduction process in fact one can also see the defect structure inside this grains are not dislocations only but also twins notably twins and this is shown in the inside picture here which shows large number of twins.

On the inside the grains so therefore theory are not only affecting the de Poisson process per se but also affecting the microstructure which can be only understood using transmission electron microscope which is having you know which is one of the best techniques available to understand the fine scale micro structural features one can also add off to this story much by using high loosen electron microscopy which will be part of the syllabus of this course here..

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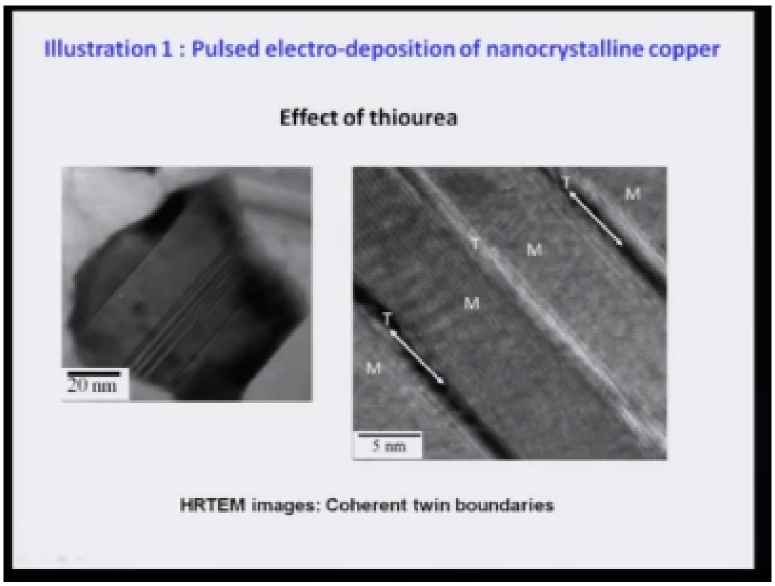

I am showing you one small grain approximately 40 to 50 nanometer diameter and if we look at inhalation mode one can see the twins which are there in the grain and the twin can also be seen here on this on this bright film micrograph on the left side a pie a slide on the right side basically it shows high lesion microscope microscopic image telling you the twins are not incoherent at that coherent twins and these are also been reported in literature these twins increases the tense strength of the material extensively.

So that the whole process as you have seen different techniques were used to decipher the different aspects of the deposition process in fact the role of theory again basically brought about by this technique this all published literature so one can access very easily from the literature so, I am not going to tell you our details of that but very easily by searching our names you can find out the literature in fact to probe that powder really affects the grain size reduction by pinning the gain boundaries of the copper grains we need to use a pretzel technique known as energy filter imaging which is normally used attached to the electron energy loss spectroscopy.

I am not going to discuss in this technique in detail in subsequent lecture, so I will not talk about the technique but I will show you the effect or use of this technique in understanding the whole process of deposition the left side of the image is basically a normal white fill image.

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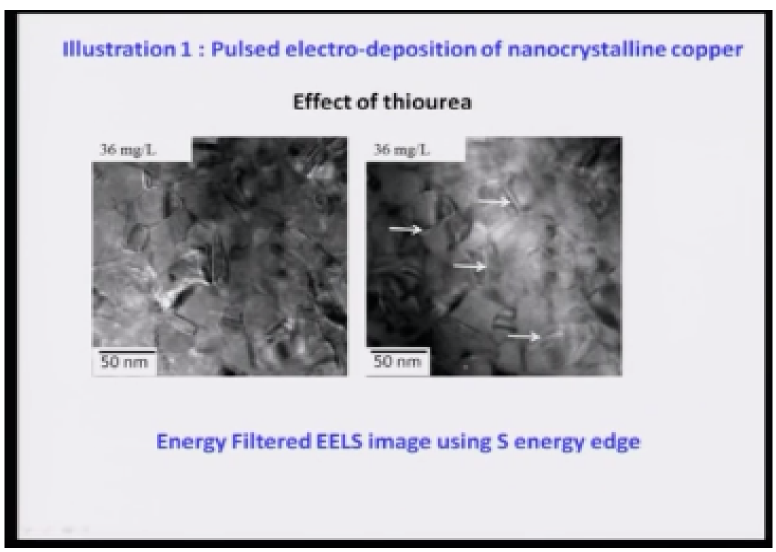

Taken by x electron micrograph microscope writes of the image shows you the sulfur energy map in fact any electron energy loss spectroscopy you can select the energies of element which are given as H energy H here we were using sulfa energy h2 image and we can see this white continues which are present along the grain boundaries signifying the sulfur. So therefore sulfur or rather thiourea is getting deposited at the gain browned is of copper and they were pinning the gain boundaries not allowing the sulfur at copper needs to go.

This is equivalent to saying that copper screens are actually capped by the sulfur or theorem molecules and that way they cannot go for the remaining inanimate regime this is another special techniques and one casting techniques ,which allowed us to understand the whole deposition process next let us station which I am going to talk about.

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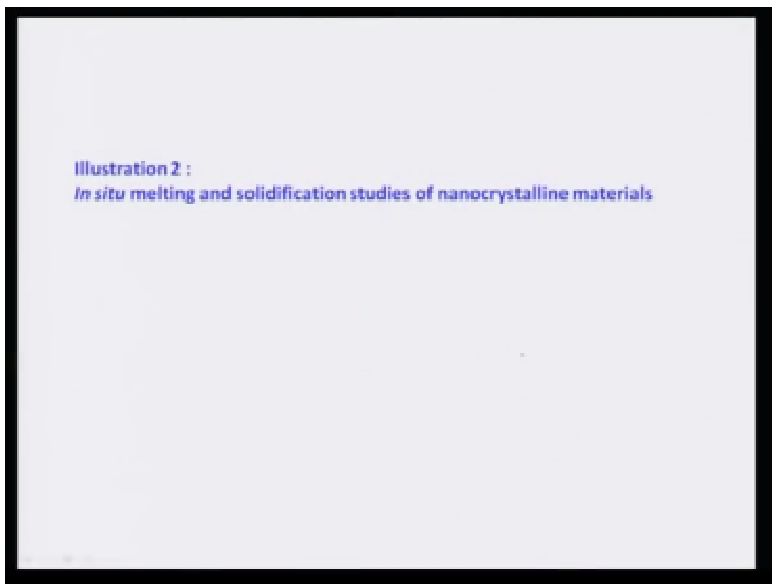

This again from our own work one of my students research work is on in situ technique you know Institute techniques are very important nowadays especially Institute inside any microscope either at an electron microscope were scanning electron microscope or even atomic force microscopes are very popular nowadays in fact there are conferences on In situ microscopic techniques so in situ is a big part of the whole advanced characterization techniques we are going to show you some sort of view that how this can be used to understand.

The phase transformation in the nanomaterials almost intimately alloy nanoparticles so I am going to talk about melting and behavior of these nanoparticles inside a microscope okay, so that you can get in feeling that an advanced characterization technique can be done the way this technique is done if you look at any time you see a little microscope those who have seen you can understand basically those who have not seen I am going to discuss with you in the next subsequent lectures.

In a tiny silicon microscope one use a holder known as a heating older so the sample can be heated up and at different rates or been specified by the by the user and can be viewed on a continuous mode like a video and from those videos one can basically obtained the transformation phase transformation taking place in the material so therefore it is a very easy to monitor easy to do a technique and in this technique one can heat up to temperature approximately thousand to fifteen hundred max so nowadays even the whole disciple level which you allow you to heat at the date of maximum five hundred degrees per second.

So definitely you can heat up the material look at what is happening by passing all kinds of solid-state transformations so this is a very advanced and very useful technique used in many material scientists to understand the basic behavior our basic phenomena taking place for any material especially used on nano materials like how the nano material grows how the nano material transformation new takes place in a system like alloy which .I am going to discuss so for this purpose I am going to say you also how different techniques can be used so here I am going to give an example on late-teen.

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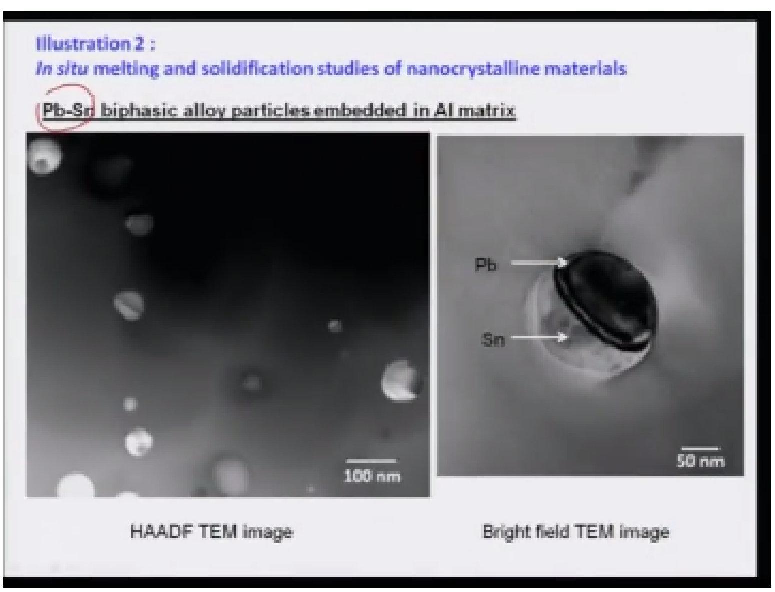

Which is very low melting eutectic alloy particles size below AL matrix nanometers embedded in an aluminum matrix this is basically prepared by root called Mel spinning in which aluminum along with late-teen alloy was melted and because lane and teen are immiscible with aluminum so therefore they there will be liquid phase separation and if you that really solidify this can lead to the small size nanoparticles the left side of the image shows you image for using hard if that is high angle annular duct feel image this is also attached with the thymus electron microscope normally this gives you images based on the contrast that is the atomic number contrast.

Just like in SEM we can get I think now my contrast using backscatter electron imaging mode here we can get at a minimal contrast using hard if image mode in fact this can be used in high elution mode also to see different type of atoms in high Lucian images but we are showing you normal white pulp image using this detector which is attached we take like ten electron microscopes what you can see here in this image is the black background which is basically aluminum low atomic number elements therefore the it will be seen as a gray or black on.

Which there are nanoparticles like this one this one or this one or maybe this one are different sizes these nano political shows a double contrast or two phase contrast one at the top one at the bottom or vice versa depends on how the particles are oriented you can clearly see the different contrasts in all the particles existing that clearly still said there are two different kinds of phases they have two different types of atomic numbers are about average at 3 numbers so therefore by using this technique one can easily map the whole microstructure using Jade G or atom number contrast if you do normal later on orbital microscopic imaging.

One can see these particles which are very small about less than hundred nanometers and it contains lead in team within the aluminum matrix where teen is the bulk part of these particle ladies forming the cap so by knowing this one can understand the morphology orientations of this particle respect to the aluminum and many other stuffs which I am not going to discuss in detail but is all available in the published literature if you just do a normal difference in scanning calorimetric catalysis.

Which is not part of the syllabus but probably your heart of it this technique will tell you if a heat or cool a sample if there is a phase transformation taking place or not here left-hand side is basically the y-axis basically power?

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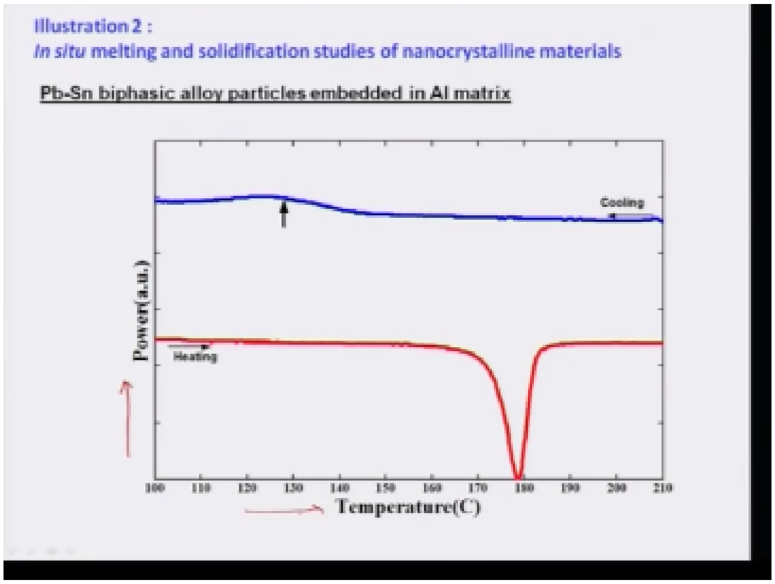

And excess is a temperature varying from 100 to 210 degree Celsius that saw the normally melts eutectic late in particles so if I heat it which is shown by red a curve you can see the melting peak which is very convincing to us there is a strong melting events but once you cool it down the solidification does not take place at a fixed temperature or range of temperature it takes plate a wide range of temperature even the side emission peak is also very diffuse so the it tells you a lot of different stops or different aspects of the whole melting stratification behavior.

Which is can we can take longer time to discuss but what do you understand in the melting is very sharp and there is a melting events before the melting event is something happens here that sort of peak it isometric so to understand it we put the sample to the sample inside a microscope or a heating stage microscope.

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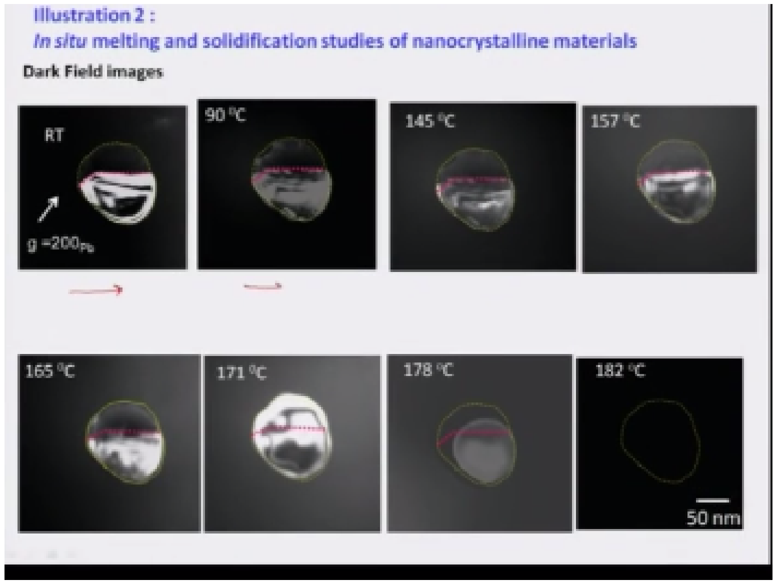

Rather and heat it up I am showing you is said of dark fill images which will tell you how this can be used to understand the phase transformation if you start on room temperature to 90 degrees or even 145 degrees nothing much happens to the particle a dark film is basically taken in such way the laid part is illuminated of the particle thin part is not please do not get confused from the earlier picture this is just to orient it so that we can understand a process and that this vector G is used to image the late part of the particle.

If he from room number to ninety degrees the small adjustment of the fade mixer takes place because of the eutectic phase diagram and then 4 again little bit adjustment the most notable thing happens at about 170 degree 1 degree Celsius temperature these temperatures are obtained from the heating hold up hitting holder we will have a thermocouple attached to it and this thermocouple sensor temperature therefore heating only cannot really sense the temperature sample but it can sense the temperature of the furnace.

So there will be some temperature difference with in a sample so but we do not bother much because we cannot measure the exact list temperature of his nanoparticle by putting a sensor which is not possible till today at 171 d Celsius temperature the whole particles become late rich so therefore a eutectic particle which is consisting of two phases transformed to a single phase particle before even it melts down and then melting begin at 178 D Celsius temperature part of the particle is already molten and I would 82-degree 182 degrees temperature whole particle is molten.

So therefore the melting temperature particle almost even same I know we know that letting you take the melts at 182 degree Celsius temperature but the whole particle does not meant like eutectic which has been reported in literature you do not need to bother about the basic things inside it why do you need to understand or I want to need to know is that the by using this institute technique we can probe even how a small sized particle.

Which is approximately 100 nanometers can be understood so therefore we need to have this advanced characters in techniques we used in a day-to-day life time to notice and this is again to show you that this is the end of the case.

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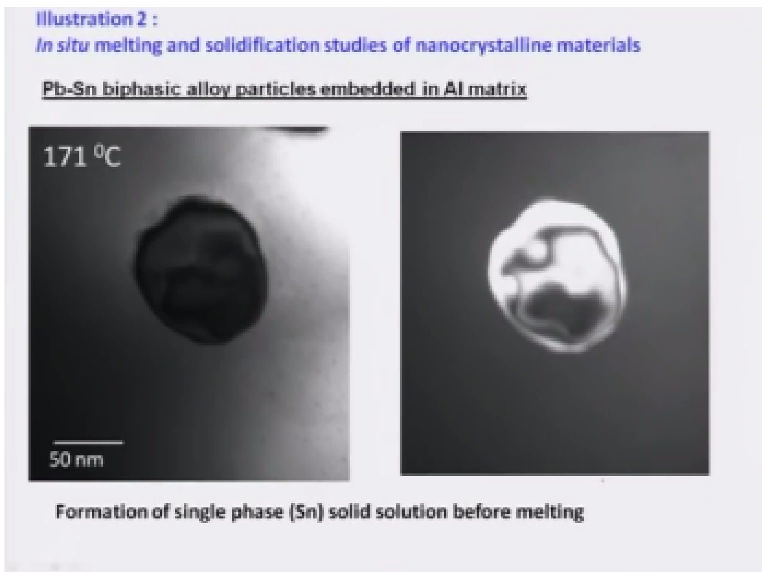

The particle transformed a single phase laid these solutions this is wrong this will be late let really solution before you when melting the third one on.
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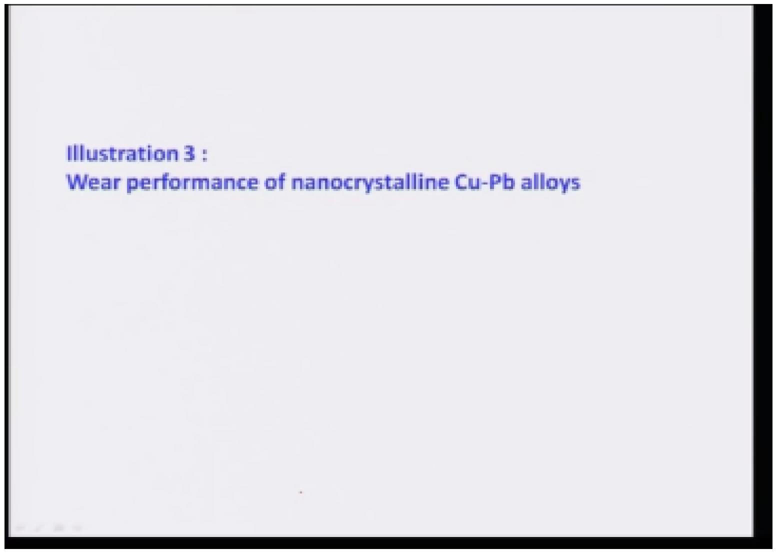

The last example which I am going to give you to you it is on we have performances of nanocrystalline copper lead who know that copper lead alloy is a very important because they used in the bearing material they are actually bearing material for many.

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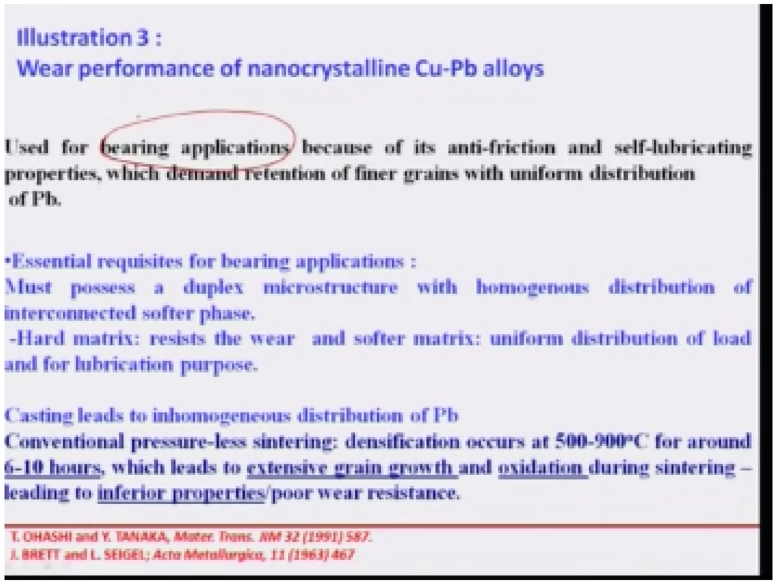

Of the objects or the machines use because of the anti friction and the lubricating properties of the lead which is normally obtained by preparing a copper late composites because ladies normally very low temperature yeah so essentially course it forbearing application is that it is mast of a duplex microstructure with a homogeneous distribution of this soft lead phase and hard matrix in which the is embedded is basically provides aware.

And the resistance and also the helps the lead particles to perform as a lubricating ones so normally this can be done using casting techniques the samples can be prepared but casting techniques leads to in harmonious micro structures many cases this also leads to some kind of late big particles of late inside the samples so that's why sintering is adapted and which we wanna use a normal simple technique it can takes you very high temperature.

But 509 a surface temperature for a long time to obtain with good sintered products so we have not used that in our study way we used a technique called spark plasma sintering technique in which the same thing can be done at three to four hundred he sets the temperature and also at a time to meet up twenty minutes so therefore one can actually form the nano crystalline copper grains structure nano crystalline process because of the use of low temperatures and so the time used in this technique is slow so these are all published in the different literature's what I am going to show you some use of characters in techniques.

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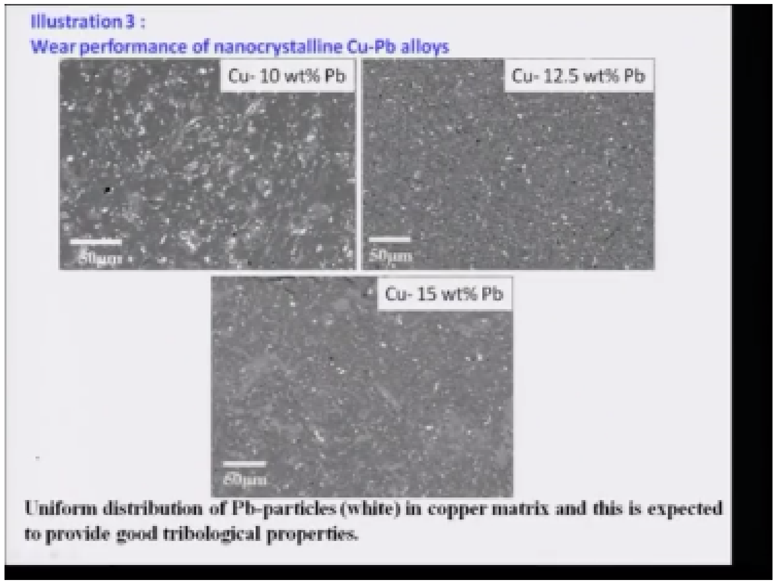

The first one is use of scanning electron microscope which is shown here this is the backscattered electron images of the copper 10% late copper 2012 and 5% late and copper 59% late and all the cases you see the white color things are late because it's a high atomic number the gray contest is from the copper and you can clearly observe the uniform distribution of late in the copper this does not tells us where the copper gains are nanocrystalline are not to lose it to understand it we go for the x electron microscopy.

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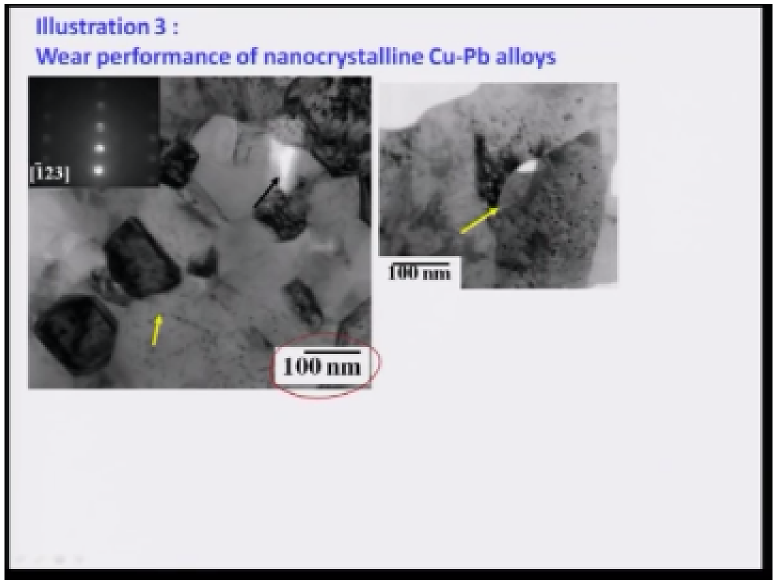

Where we can see that these greens are indeed approximately one hundred two hundred less than hundred or even some girls hundred twenty nanometers and late can be seen between the two gain of the copper or at the conjunction point tipple junction points one can observe even lead particles which has fallen off during team celebration so these two techniques using scanning tunneling list of microscopy and also times microscopy we can see both the Cu-Pb alloys distribution as well as the copper grain size.

Now we feel I understand it will obviously it has a very good distribution of will clear in copper with the Nano green copper will have very good friction properties that is what has been done in this case you can see the coefficient of friction.

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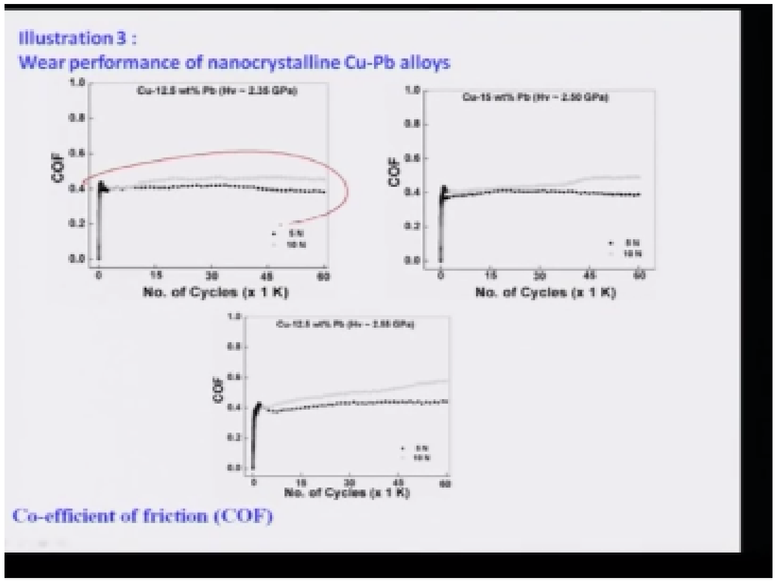

Can be as low as point 4 in all the cases and by showing it we can say that the frictional property of the material has improved but how to understand that.

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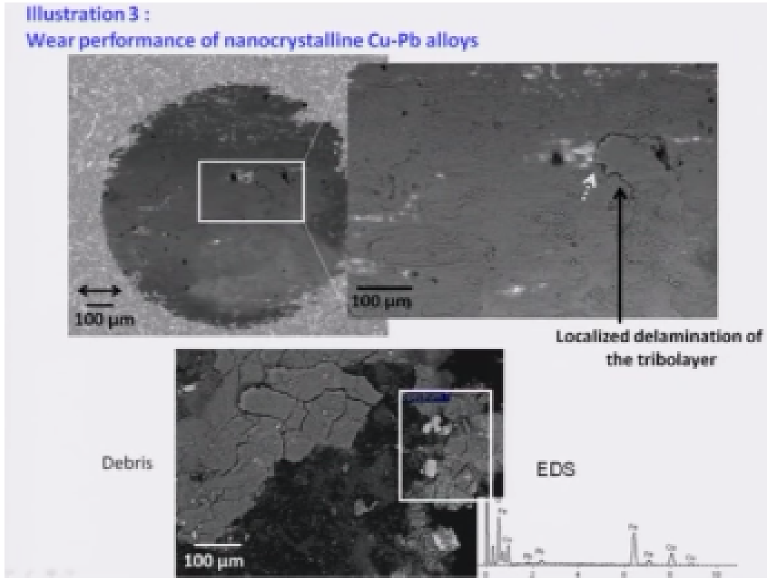

We have looked at the wear surface you known as SEM or scanning electron microscope one can see different kind of things one can see delimitation one can see even the breakage of the particles by using this character microscope even if you use the GD ax that is the electron spectroscopy energy dispersive spectroscopy like by using electronic character microscope we can see the peaks coming from iron oxygen copper laid.

Even iron is very predominant because we used the iron ball or the steel balls for a braiding the surface so iron comes into picture and from centavo layer not only that to even understand that how the oxygen is affecting the microstructure where process we can use Raman spectroscopy.

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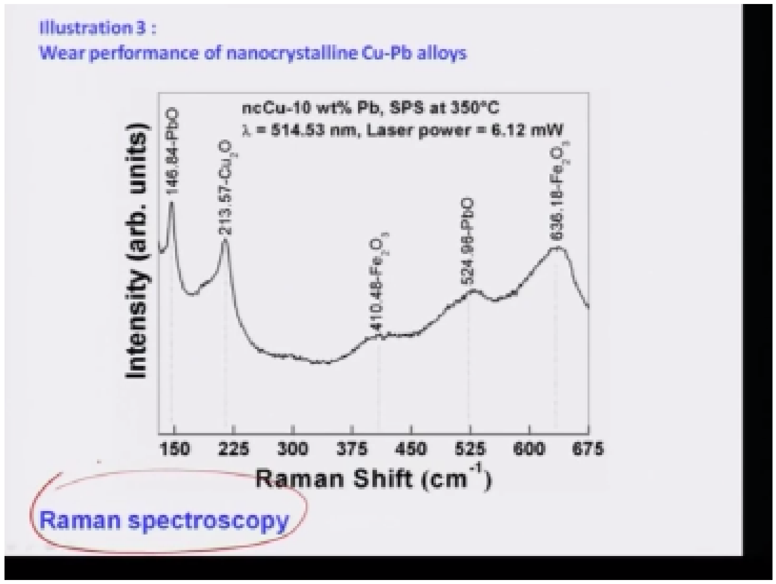

Very small sized laser beam can be focused on the sample and the dominant peaks can be obtained this source of presence of ncu-2o a few 2 or 3 lead oxide on the surface therefore oxidative oil takes place on that.

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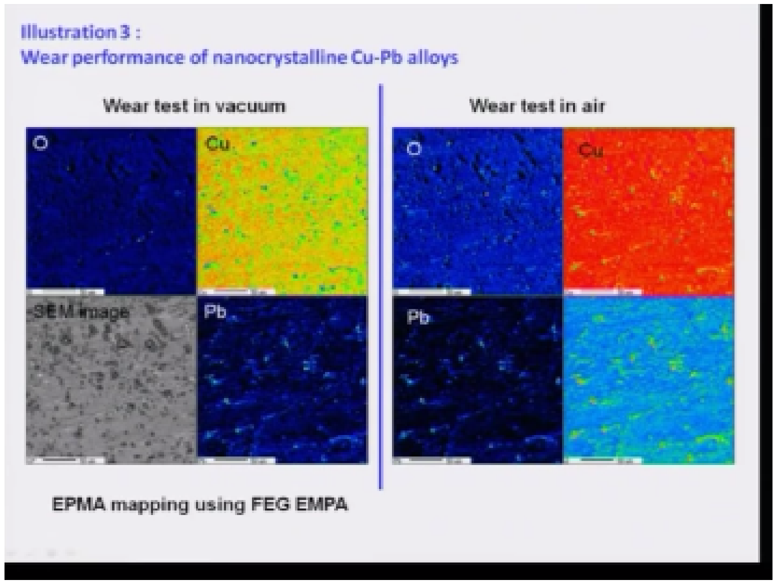

To clarify it even one can do vacuum where taste and then map the elements using electron microprobe analyzer if you see that the oxygen presence on this sample which is tested in vacuum is very small on the other hand piston in air oxygen signal is very high the other signature copper and late so which openly present predominantly on the sample this is another important techniques like electron microprobe analyzer which is using a pestle gun like focus the field emission gun can give us a very high resolution.

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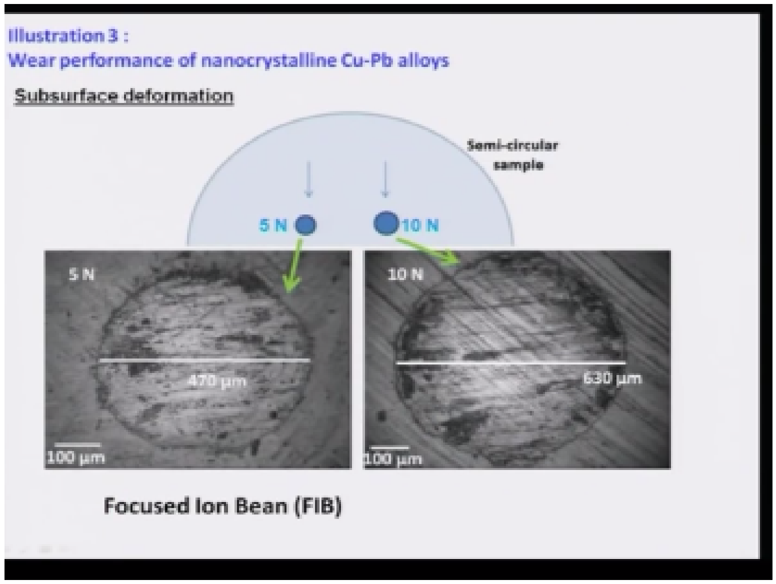

Then one can actually look at subsurface imaging use focused and beam in this technique sample is dig.

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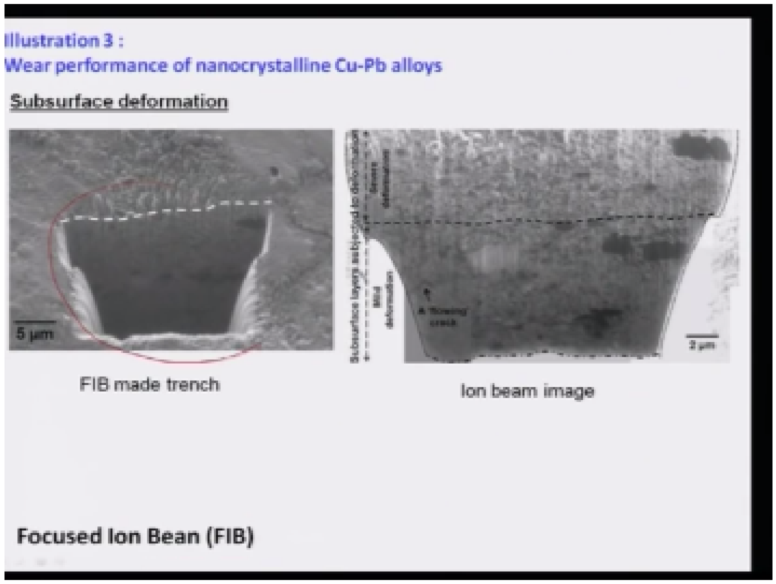

A trench is made and a sample and the surface inside this trench is seen either on iron beam to show the cracks or the different particle present all time.

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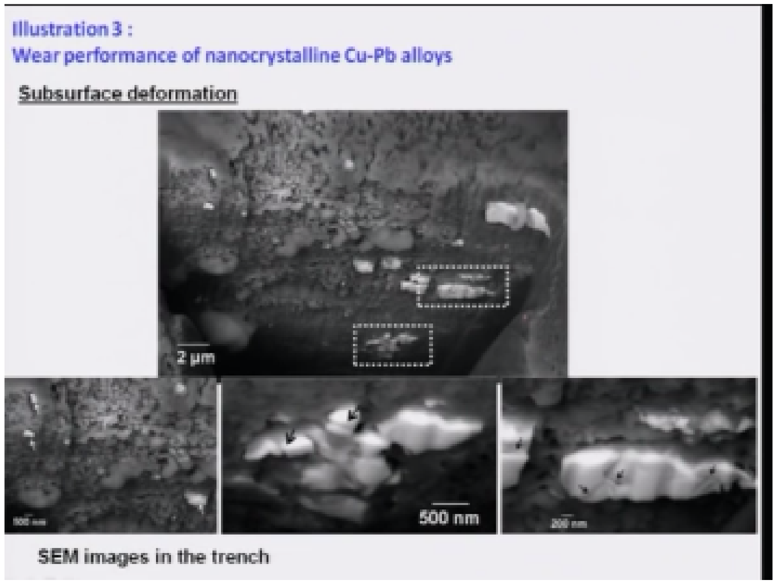

You select on a scanning electron micrograph like that where you can see these copper cranes or coagulated copper games lead particles even some of the lead particles can be seen to be fractured steps to be formed and some cases they are sintered you can see some cases there sinter actually here so therefore all kinds of phenomenon a phenomena can be easily observed if you look at the subsurface imaging using focused ion beam this is again shown here how different particles.

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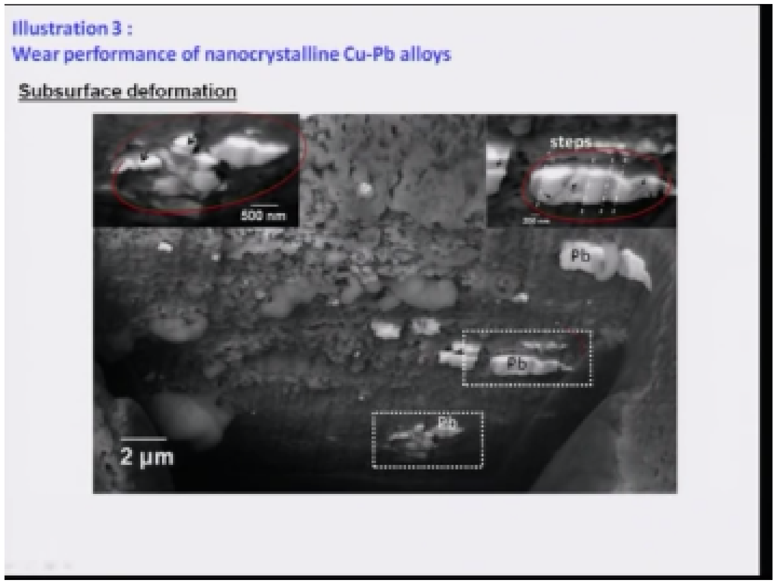

Has been fractured steps as form of sinter.

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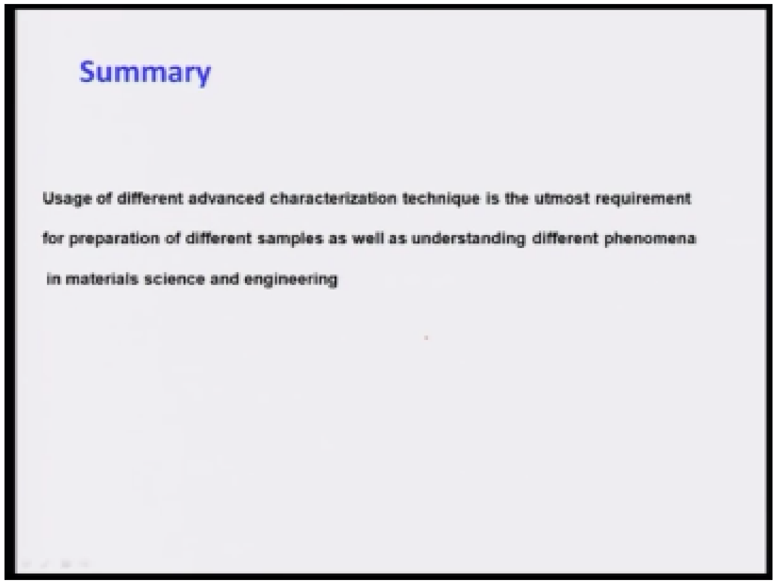

So in summary what I can say you is this that we need to use different advanced characterization techniques to understand the phenomena taking place during processing.

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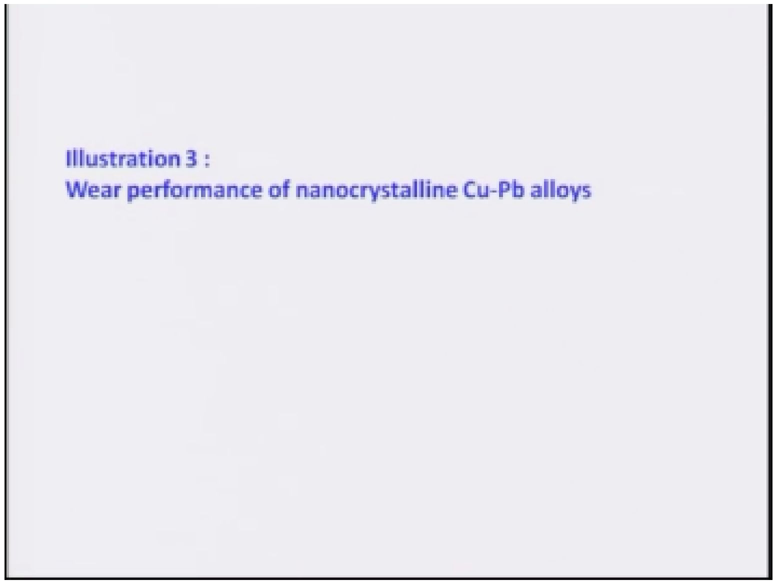

Third illustration or the last one in this class in this lecture is on we were performance of nano crystalline copper ladle ice we know that copper lead alloys are used extensively in bearing applications and this is done for long time because of the anti friction properties of plate also self lubricating properties.

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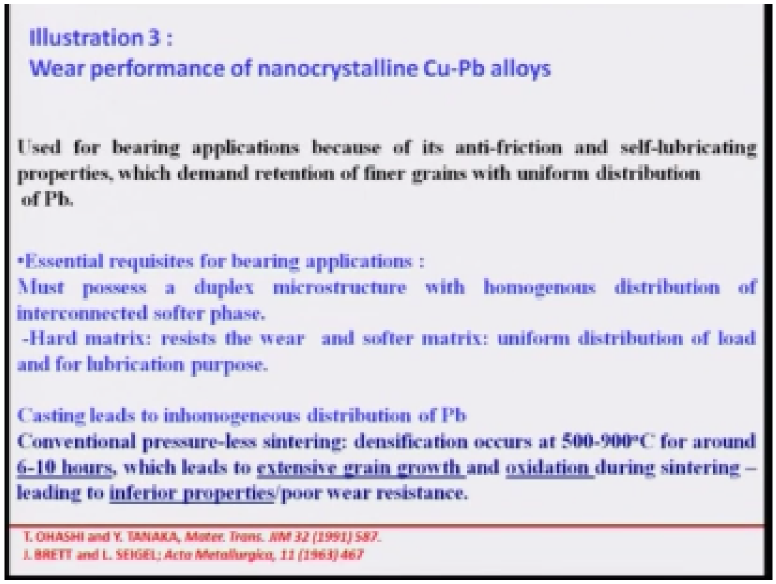

Of the lead within the copper and later immiscible in actual sense in solid and liquid state so therefore they form very nice distribution of the laid in the copper normally for any bearing applications we know the material must have the following requirements as far as the microstructure is concerned it must have a duplex microstructure we know that for bearing application the materials must have following micro structures or requirements first of all it must have a duplex micro structure consisting of homogeneous distribution.

Of the second particles like lead here in a matrix Cu-Pb the matrix actually resists the we were becoming harder and the soft phase which is the lead here which can act as a lubricating agent thereby reduces the wear and tear so it has been seen that copper lead our lives can be prepared by casting roots and this roots leads to you know when your distribution of the blade in the copper that is because lead immiscible with copper and that is why the distribution of the leading copper will be very bad because of the different problems in the casting techniques that is so no one uses normally the casting rule to prepare Cu-Pb alloys.

Rather use sintering techniques or the powder metallurgy techniques in the kinetin sintering routes where copper and lead powders are mixed and then sintered by persona list technique it has been observed that it takes about 500 to 900 Celsius temperature by heating the sample or the powder mixture for about six to ten hours to get dense center product and this temperature and the time obviously will lead to extensive grain growth of the matrix of copper.

And also it can lead to oxidation of the both copper and lead so that is why nowadays one can use pistol techniques to stop the grain growth for keeping the nano crystalline copper grains in the microstructure at the same time to stop the oxidation both are required for better performance of the material this can be done by using a pestle technique known as spark,plasma, sintering, technique.

Which is a very recent one which has been developed in 90s and hardly about twenty years old so this can allow us to use lower temperatures of three to four hundred Celsius temperature for sintering a Coppola delays and the whole symptom cycle can be finished within ten minutes so therefore it will stop the grown gain growth because of low temperature in time and also because of this time at high temperature oxygen can be reduced in our case we have used this techniques.

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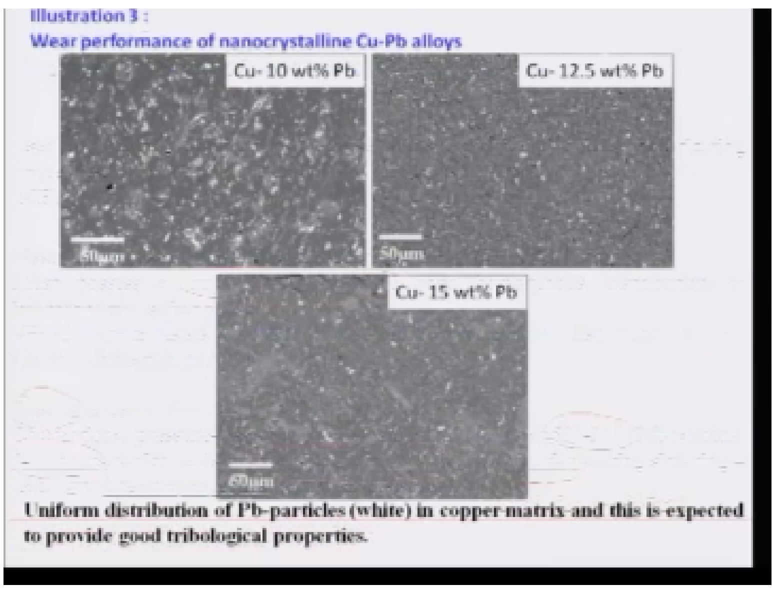

To prepare the samples then we have analyzed. The sample using different characters techniques so that we can understand where they are of this material first. I will show you the initial microstructure here. I am showing you the microstructure of the copper laid alloys with different lead concentration ten twelve point five and fifteen percent blade whiter them see is this white faces some cases they are discontinuous.

And some cases their particles they actually led this images are obtained in scanning electron microscopic image using basket electron mode which you will discuss as a part of this course which can show up the Jade contrast or the item number contrast lead having the higher atomic number will be seen as a white say ladies uniformly distributed in all the samples as you can clearly see and copper can be seen as a great contrast. But grains of the copper cannot be seen in SEM they are very small to observe the grains of the copper.

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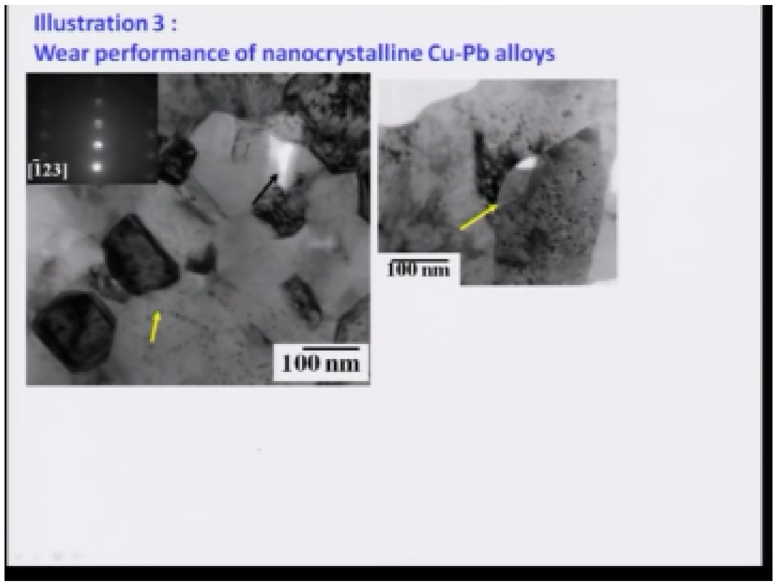

We need to see the samples in Tanis electron microscope and these are the two images taken from an electron microscope or the copper late sample you can see the grains are very small this is the honey nanometer bar so approximately hundred two hundred twenty nanometer some cases even lower than honey nanometers grains can be seen and laid particles are seen to be situated between the copper grains many cases between the typical junction points of the grains or some cases red particles has fallen off during team separation.

So therefore by using both the scanning Teresa lacto microscopy and the times electron microscopy techniques we can understand the microstructure very well and by seeing the microstructure you could probably understand that this is a very good material for where registered study this has been done using fitting wire technique and here I am showing you some.

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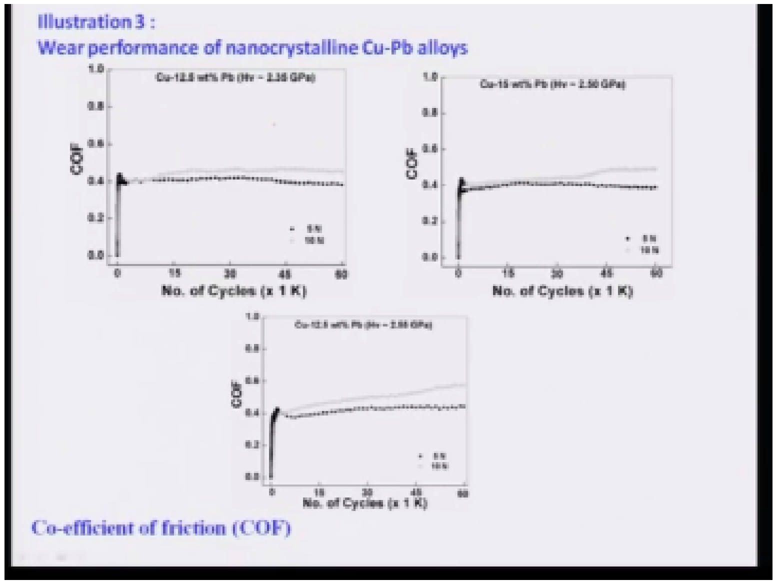

Coefficient of friction values obtained by our study you can see that for different concentration of the late twelve point five fifteen or this is also twelve point five but different hardness so what you can see that if cushion friction is pretty low as compared to copper coefficient friction of copper is about 0.8 again steel and this wear test is done using a steel ball on a fitting wire machine so the because of that the coefficient of friction is because it is low that means laid as a prominent role reducing.

The coefficient of friction not only that the hardness of this material is pretty high as you can clearly see because of the nano crystalline copper grains.

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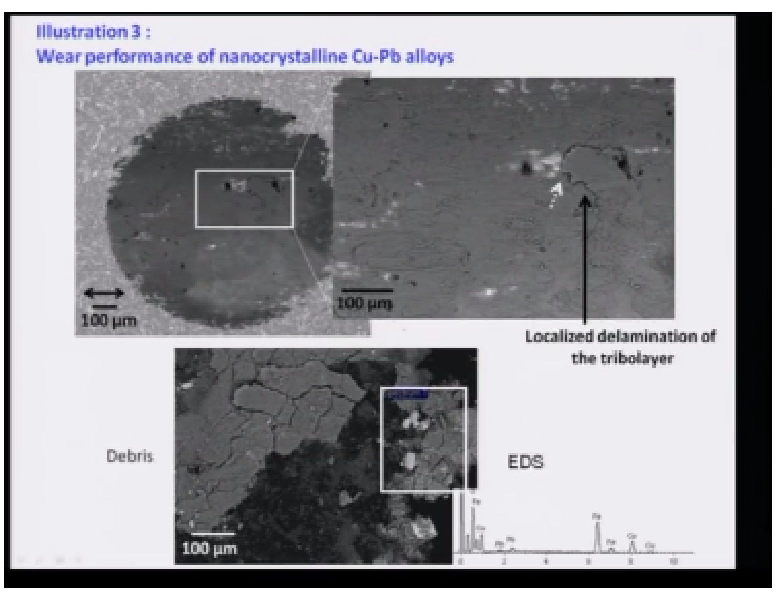

So now how the characterization techniques can help us understand a wear process here I am suing with a West car during the fitting we were at a low magnificent picture which is approximately about several millimeters whether this is 0.1 millimeter so therefore it will be approximately several tens of millimeters and with him that if you just look at in zoom view you can see delimitation you can see even cracks some cases you can even see the lead particles sprayed across the sample.

So if you take a lead ax of this surface the DAX means energy despite a spectroscopic technique attached to a cm we can see presence of oxygen iron copper obviously and laid iron is predominantly present there this is because iron is getting transferred from the steel ball oxygen is also presents therefore what is the exact role of oxygen one needs to understand very clearly that to do that we have done Raman.

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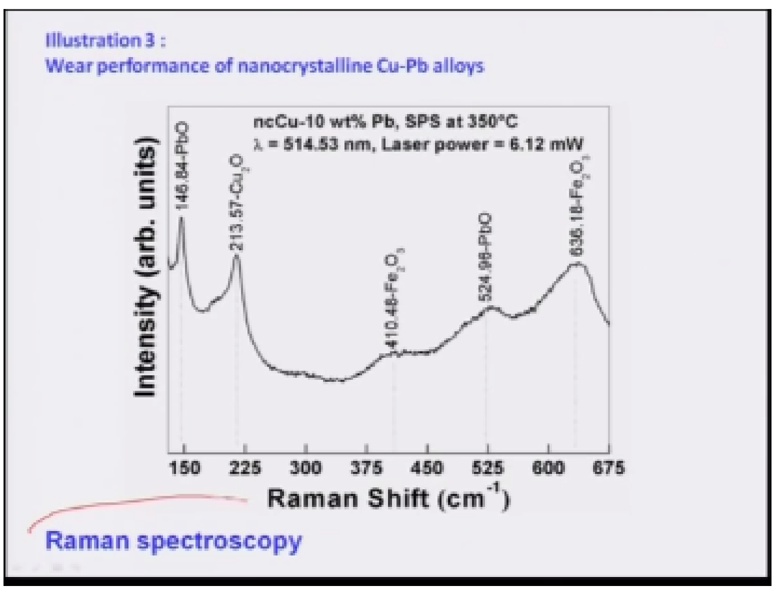

Spectroscopy on this wear sample or the own surface if you do that using a laser beam of 540 point 5 2 nanometer. They are very small laser power one can see presence of lead oxide copper oxide iron oxides on the surface therefore oxidation do takes place on the surface because of wear.

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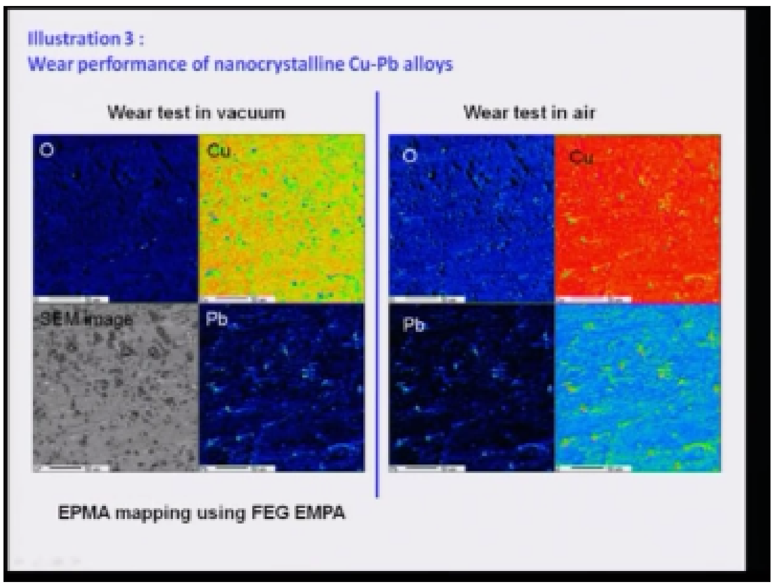

But one can easily prove it even using a better technique known as electron microprobe analyzer here we have done tests where test in vacuum by testing air to see the oxidation if you use electron microprobe analyzer in a fag EPMA that is the preliminary period the beam size is very small the solution will be better so you can see the oxygen is very small even on the surface for the sample tested in vacuum on an oxygen is quite large in the sample test study in the air copper and lead are obviously present on the sample and this is the SEM image of the sample on.

Which this EPA analysis is done so therefore by using these two techniques gammons and the PMA we can clearly see how the oxidation do takes place oxygen gas takes place in the where process and changes the wire kinetics do you know how to look at this subsurface deformation characteristics one needs to use another kind of characters in technique.

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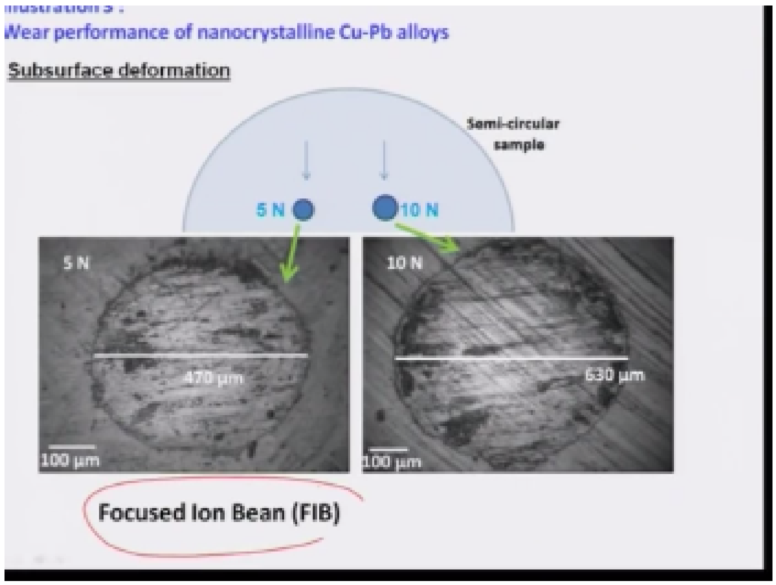

Which is known as focused and beam to prepare the sample so what do you do take this wear track and cut in middle and make a trench we can do on a different actually wear surfaces this is so in the next figure.

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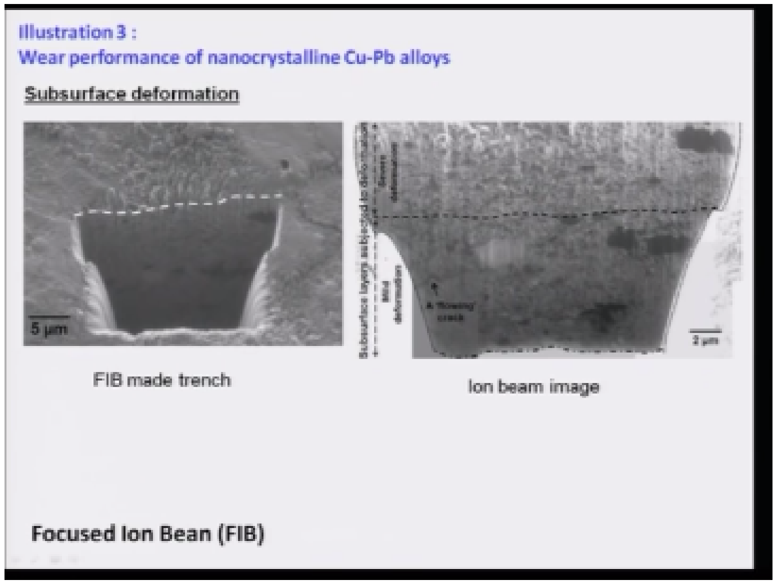

Here we have made a trench tracked using focused ion beam or the gallium beams in the focused and beam machine and if you just steal the sample in pew even in the focused and beam by using the iron beam contrast one can see even different black color things which are basically particles you can see even they have cracks which is going like this better image can be obtained.

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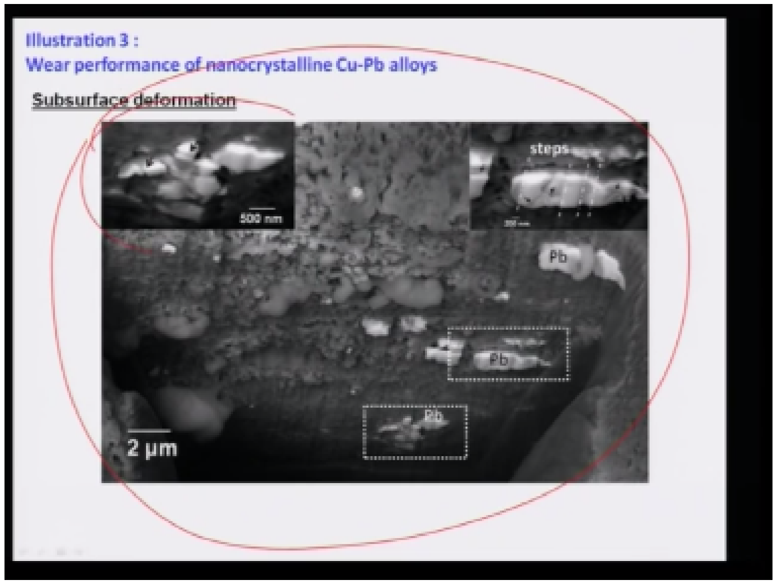

If you see this trench in scanning electron microscope you can see the laid particles peak white color and also the copper agglomerated grains lades has seemed to fracture in many cases some cases steps to form on the surface of the laid and some cases relates a small size particle some cases they accumulated because for sintering so therefore during all wear process temperature do rise in the subsurface domain of the sample and leads to the sintering and during deformation some of these lead particles do gates deform and steps forms on the surface.

And fracturing also do happen so this is again shown here in a very high magnification picture within sets so this you can see very clearly so what I am trying to convince you is that by using this set of characters and techniques which are known as advanced characteristics we can see this face of surface of the sample during wear process and understand the wear mechanism very easily see in summary and also for the subsequent lecture.

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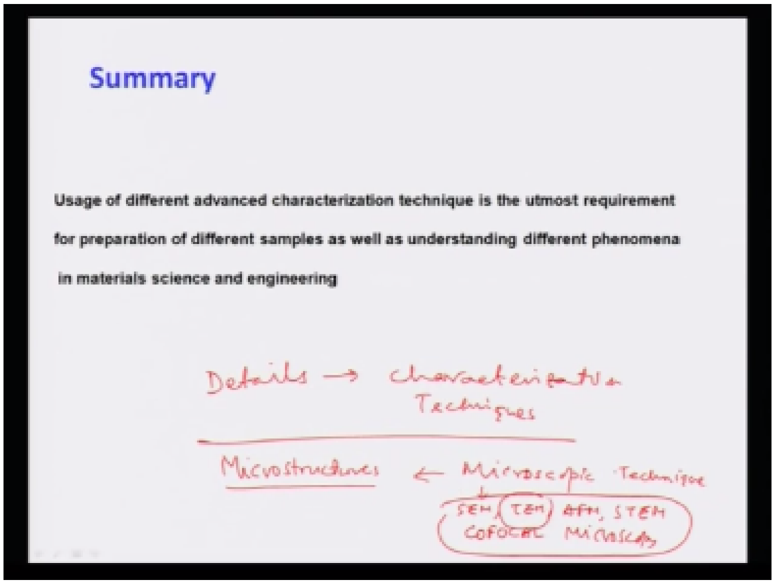

I am going to say you something here that in some way I can say that we need to use advanced characters in techniques for not only to understand the material science phenomenon in a particular process but also to understand the processing techniques which are used or required for the whole you know material science domain many cases we need to engineer the material and in nearly material means isn't changing the processing parameters change sample characteristics temporal compositions how does these parameters affect.

How do this parameter actually affect the processing are the material properties later on can only be disulfide if we look at the samples using different characters and techniques and this character in techniques can be either microscopic techniques as we have seen or you can respect scope techniques many cases we need to use even diffraction techniques to understand the processes which I am not showing in the first lecture or many cases even, one can need to use the surface characters in techniques like the Aussie electron spectroscopy are any other spectroscopic techniques which can give us surface properties.

This is more important for the particles which are very small size like nano size or even some cases micron size so therefore in a nutshell what I can say is that that we need to know in detail the detail characterization techniques which are used or which will be used by many of these labs for understanding the material science phenomena. So in the next class I am going to start with microscopic techniques will be used to understand micro structure.

So therefore the connecting the micro structure with the material processing can be only done by using different microscopic techniques this will involve SEM, TEM, AFM, STM as well as Confocal microscopy we will discuss one by one these techniques I will first start with TM in the next lecture then moving to SEM and finally other techniques will be dealt with thank you.

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