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> **Lecture No. # 05 Experimental Tools & Techniques**

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Good morning. Today, we start a new topic in this course, and here in this particular chapter, we will learn about a number of tools, and techniques which are used to examine the structure, and properties of materials. For example, in the previous chapter, we talked about crystal structure. Now, the question comes how do you know that metals are crystalline. We talked about lattice parameter, but how do we measure this lattice parameter. So, we will come to know some of the experimental techniques which help us to know number of properties, and the structural characteristics of materials.

In this course in this chapter, what we are going to look at primarily, the tools which are used to examine microstructures of solids. Often I mean that is termed as Metallography, but in practice it is actually the micro structural examination of solids. And we have different types of microscope, that we will talk about these are optical could be transmission electron microscope, scanning electron microscope.

We will look at the main features of each of these, then we will learn about X Ray Diffraction - this is the technique by which you find out lattice parameter of crystals. We will know about number of Mechanical Properties and what was mentioned, it is the mechanical properties which is most sensitive to the microstructure of the material and there are number of mechanical properties which are of great importance and we will look at some of these and also to understand how structure develops in solids it is often necessary to know about Thermal analysis. Now, I have sequence this lecture not exactly in this sequence. I will first talk about thermal analysis and then come back to metallography, X ray diffraction and possibly next class we will talk about mechanical properties.

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Now, first of all before we start, I mean the question comes why do we why is it necessary to know about thermal analysis because most of the structure that forms in solids you know it develops from the liquid when it is cooled and when crystal forms in the liquid different types of structures they develop and in order to know the kinetics and thermodynamics of this transformation, that is necessary to know a little about thermal analysis and here one of the key tools that we will be using is the thermocouple which is a temperature sensor.

So, let us say what is a thermocouple it is a temperature sensor temperature measuring device. It consists of a pair of metallic wire joined at one end which is shown over here. This is a metal A, let us say this is metal B and they are joined over here and if you heat this point, this particular point if you keep it in a furnace at a temperature T and we call this as a hot junction and and then we take the output from this end which is maintained at a room temperature all we call it cold junction and we connect it to a milli Voltmeter.

We will find that depending on the magnitude of this temperature, we get a particular milli volt reading in the meter and this is a very convenient device for measuring temperature and the milli volt that we get, is a function of the temperature difference; that means, temperature of the hot junction minus temperature of the cold junction and in ideal cases we will prefer a material the thermocouple where this relationship is linear, but in reality most cases even though it is nearly linear, but may not be perfectly linear something like this the milli volt reading and the function of temperature is shown over here.

So, this is the characteristic of the thermocouple and here in order to measure the temperature suppose we get a certain milli volt reading say this is the magnitude which is shown by the meter. Now, since this milli volt is proportional to the temperature difference and this plot may not be perfectly linear, the way of converting it the temperature is illustrated here, what he would do to this milli volt you add the milli volt corresponding to the cold junction at this. Let us say for the room temperature, this is the milli volt if you add the two then this is the total milli volt and what you would do, you read the temperature from here. Now, the question comes what are the thermocouples commonly used thermocouples which are listed here, one is the chromel and alumel.

So, one of this say this is positive; this is negative and this thermocouple this is primarily an alloy of chromel, is an alloy of nickel and chromium, chromium and nickel this also is the chromium nickel with a little bit of aluminium and this is used up to a temperature I mean quite a wide range and if you want to go to higher temperature platinum, platinum thirteen percent rhodium is commonly used beyond an between the two this is more linear corresponding to that.

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Now, let us look at what is thermal analysis. In a thermal analysis, what we do say suppose we heat a piece of metal which is kept in a furnace, and we have a thermocouple put into the metal say can touching the metal $(())$ here, and this thermocouple is connected to a temperature monitor. And you heat the furnace I mean you supply power to the furnace, and what we do we monitor the temperature of this piece as a function of time. And which is shown over here, the temperature as a function of time.

As long as, it is solid you will find the temperature goes on increasing and that when it reaches its melting point then we will find that temperature remains stationary until the entire amount of solid is converted into liquid and once it is converted into liquid again the temperature starts raising, but usually the heating is more difficult to control because it is involved as we know lot of heat transfer and other features which are involve the thermal mass of the furnace and lot of other things and it is often difficult to control the heat I mean get a good time temperature at time temperature time plot in case of during heating whereas, during cooling you get a much better information because what you do you switch off the furnace and let it cool slowly and monitor the temperature and then which is shown over here and then this is the melting point that information that you get from the thermal analysis is the transformation temperatures.

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So, this is a case where a metal, I mean for a particular metal, pure metal we find its melting point, but we have much more several more much more sensitive equipments for thermal analysis and these are well instrumented. Particularly, say one which is shown here is differential thermal analyzer.

So, what he would do you take a piece of that $(())$ sample. This is my piece of the sample over here which is put in a container and you also have a reference material. Now, what he would do you put two thermocouple and you connect them in the opposite way. Say let us say, if the if this is the negative, you connect this to negative and then the positives are over here. So, what you are actually measuring is the temperature difference between the sample and the reference, but mind you this magnitude is very small you will need an amplifier to magnify it and what you do, you note down this reading the difference in temperature.

And also you have another thermocouple kept in between which monitors, the temperature of the environment and both goes to the recorder and what you plot is a this temperature difference between the sample and the reference standard against the temperature of this environment or surrounding or basically you can say it will be further practically the temperature of the reference material. So, this is and this is the type of plot you are likely to get and this delta T which is shown over here and depend the T S minus T R and what you get is shown here.

As you raise the temperature say one, say suppose some endothermic reaction takes place or transformation takes place then what will happen; that means, some transformation is taking place in the sample its temperature will not raise, but since the power supply it is power is being supplied at a constant rate, the temperature of the reference material will continue to increase. So, then this becomes higher than this. So, this is negative. So, you get an Endothermic peak. So, likewise there is a possibility if there is a transformation taking place here which is Exothermic in that case, you will get a peak in this direction.

So, this $($ ($($)) compare to the normal thermal analysis, differential thermal analysis gives you little more information it gives you some qualitative idea about the heat effect and thermal properties. In fact, this area under this peak will be a function of the latent heat of transformation say we call it say let us say delta h may be it will be a function of the amount of material that you have, it will also a function of some thermal characteristics of the material, let us say specific heat thermal conductivity etcetera and it is seen that the sensitivity of this type of measurement increases the peaks are become sharper, smaller if m is less.

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Now, there is another variety of variation of thermal analyzer which is called Differential Scanning Calorimeter. Here, instead of having a common power supply you will have two independent power supply here, also you have a reference material and a standard material that sample and this is a reference material usually reference material is. So, selected which is very stable does not undergo any transformation in the temperature range you operating the system and this is the sample and what you do, you connect it in the same way as in the Differential thermal analyzer the D T A and here also these thermocouples they are connected in an opposite fashion and the difference of the temperature difference of the temperature between the two is fed to an amplifier which amplifies it.

And it sense that $($ (
) that value to a controller and what the controller does, controller tries to control this temperature difference. It will try to keep it 0 so that means, it has to supply power supply is the differential power supply different amount of power will be supplied to the reference material and the sample and. In fact, what you are going to measure the amount of power that you are heat that you supply to the sample and the heat that is supplied power that is supplied to the reference material and this difference per unit time is plotted in the y axis and the temperature of the surrounding is monitor by another thermocouple which is fed to the recorder and you generate a plot, plot of this heat effect that amount of power which is being supplied.

The difference will power that is being supplied does a function of the temperature in the surrounding and here again you will get a similar that endothermic connects to thermic peak, but the sense it will change. This is because an endothermic peak here, it will be just opposite of the differential thermal analyzer and here since it is directly proportional that heat that heat effect is directly proportional to the power which is differential power which is being supplied. The peak area is proportional to the latent heat of transformation and it is not a function of thermal characteristics like specific heat and thermal conductivity.

So, in short this definitely is much more powerful than differential thermal analyzer and it can be take very small amount of enthalpy change or latent heat and some of this instrumentation you know that they are also couple. Sometime along with that there is a load cell connected. So, you can also monitor the change in weight as well, but let us not bother about it. So, I think what is important why I spent some time on thermal analysis because very often to explain might how the microstructure evolves in a material. We will we looking at the cooling curves and how the transformation takes place. You can get the lot of informations from D T A and D S C plots. Next we will come to has been mentioned.

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So, once it is solidified you get a solid mass and we mention number of times that this metallic solid it is crystalline and how so that means, is it the entire mass, is it is one crystal or is it made up of multiple crystal how do we know. So, normally for this what we use is we look a structure of a metal and a microscope and what we do we take a piece of metal cut it, we mount it in a plastic, it could be a cool setting plastic or we can mount it in a Bakelite and then we polish the surface because metal is not opaque. We have to if we have to look in a microscope, we have to look it look at it in deflection mode and for reflection to occur, the surface should have a $(())$ finish.

It should be shining. So, there are number of steps involve in the preparation of metallic samples for micro structural examination which is listed here, a cut or mount then you polish just mechanically polish the grind and polish on a emery paper there are series of emery paper with a different a great sizes and finally, you polish it on a cloth this is a $\frac{1}{2}$)) polishing usually and where you have water and polishing powder mixture which is put on a rotating disk, where you have a cloth mounted on it.

So, this process is called lapping and after that what you do, you etch this specimen say normally the polish surface, you put some chemical agent aging reagent which will attack certain areas of the metal and then you put it in the microscope. So, which is particularly shown here, Microscope as a lens a microscope this is an objective and this is the metal piece and when you etch it is no long smooth initially it is smooth when you polish it designing, but when you etch it number of certain areas dissolve more than the rest and which is shown here schematically and when the light falls on the sample part of it wherever this phases are inclined part of this light they get reflected and.

So, whatever you see through the eye piece is not the entire light part of it. So, which is systematically which is schematically shown here say from this part the entire light it goes back and here, I have put it as a dot. So, basically it will show that it is the brightest area and from this region lot of light gets deflected out. So, less light is coming from there.

So, this will have a different contrast. So, what you get is a deflection contrast and what you find this metal is made up of the it will appear even if it is a pure metal; it is made up of several crystals. Shall I this is one crystal I mean this is another, this is another. So, they are made up of several crystals and which we normally called metallography grain and extend of etching depends on the orientations. So, what we call it some oriented grinders, it is a differently oriented grind gives different luster and from this microstructural examination we can get lot of information about the grain size, the shape etcetera. In pure metal whereas, in a more commercial alloys there will be multiple phases.

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Now, this is a typical construction of an optical microscope as I said that, metals are opaque you have to examinate under deflection mode. You have a light source and you have a lens which makes this light from this parallel, there is a mirror this is a half silver meter part of this light is deflected and it is through an objective. It is focused onto the sample and the deflected beam also go passes through the objective and passes through this meter and here you have the eye piece or you have that image plane where the image is formed. You can put a photographic plate or a camera can be mounted on this picture or the microstructure of the sample can be recorded.

Now, the question comes what is the resolving power of a microscope. Resolving power by resolving power we mean, what is the ability of the microscope to clearly see two very closely spaced point which is shown over here, suppose this point and here can we resolve them clearly will see them as distinct constituents or distinct points and this distance this distance is lamda. It is a function of the wave length that we used to examine the microstructure and optical microscope what we use is the visible radiation and visible radiation has wave length as you know around four thousand to six thousand angstrom and this resolving power is equal to the wave length over twice numerical aperture.

This numerical aperture means, amount of the light which comes and which it is $($ ()) of. So, basically this numerical aperture is a function of the refractive index of the medium here, this medium. So, in most cases it is here, let us say and it is also function of this angle this particular angle. Now, the maximum angle this angle, the maximum angle it can be ninety $(())$. In that case, this objective almost virtual you will be touching the eye piece. So, this is the limit. So, if we say that it is possible to reach them. So, the in that case, we can say that this is one.

So, basically this resolving power has a direct relationship with the wavelength that we use. So, resolving power approximately we can say in this refractive index of a is one this is one. So, we can say it is of be order of approximation of the order of lamda over two. So, if we use the smallest wavelength. Now, the violet region we can expect the two thousand angstrom to be the resolving power of the microscope (No audio from 25:40 to 25:47)

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Now, the optical microscope although we get an idea, how the difference range in an optical microscope how the different grains are arranged in a solid, but in present we get from optical microscope, but optical microscope will not give us the crystal structure, what is the crystal structure of that particular grain because metals they are deformable they can take any shape, they can be made to take any shape unlike rock where there are different crystal phases are quite prominent. This is not so in metal and in fact, that the metals are crystalline first experimentally cooled only when X- Ray Diffraction technique was discovered. And X- Ray Diffraction, what we use is an X ray wave, an X ray is an electromagnetic wave.

It has a very small wave length as we said that, resolving power is a function of wave length in case of an optical microscope. In fact, if this if this wavelength becomes very small, it is possible to resolve even the crystal structures of solids and. X ray have it has a very high penetrating power also. It can pass through thin pieces of metals. In case of steel it will be a thin, but in case of aluminium even quite thick samples of aluminium. X ray will able to penetrate to and how do you get X ray basically, it is a that $\frac{it}{it}$ is a tube which is a $(())$ vaccum tube $(())$ vaccum and you have a filament which is heated and if you heat the filament, you get electrons emitting from the filament and this electron is accelerated to a very high voltage of the order of several kilovolts the 30 or 40 kilovolts.

So, but you will have will have a stream of electron moving in this sealed tube and then it strikes a target. This targets are made up of metals and which are water cooled and from this metallic targets you get X rays emitted because what happens this beam of electrons has high energy and when this electrons strikes this looses that energy and part of this energy is emitted as X rays. An intensity of X rays that is distribution is shown over here, this is intensity as a function of wavelength you get this type of plot you there are certain peaks also which are noticed, these are characteristics wavelength of corresponding to this metal target. So, this are called K alpha and K beta radiation.

And this you must have read in physics, these are the characteristic emission from a metal when they are excited because we know that electrons, metals they are moving in different orbits which like K L M N. Now, if this electron which is striking the metal target removes an electron from the K shell then it becomes and it goes to an excited state. It is unstable then electron from higher orbits say L drops down to K and in that case you will have K alpha radiation emitted and if an electron drops from M to K, you will get K beta radiation. Now, the this wavelengths of this characteristic wave length this is a function of atomic number and very often an X ray diffraction.

We use some specific characteristic radiation like copper radiation; copper K alpha has a wave length of 1.54 angstrom. Similarly, we also use chromium K alpha a molybdenum K alpha radiation to record diffraction pattern and $(())$ in other cases we also use the white radiation to record diffraction pattern from single crystals and in case of white radiation will prefer usually tungsten target for monochromatic radiation. We will use only prefer the characteristic radiation from certain specific metals like copper, chromium, molybdenum etcetera.

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Now, X rays they are electromagnetic radiations they have very small wave length and now imagine, if that takes place is incident on a crystal which is represented over here as a regular $((\cdot))$ atoms represented by these points and these are two crystallography plane. Now, if this beam is incident at an angle theta, this is the incident angle. The angle of reflection is also equal to theta this is the reflected beam. Another beam which penetrates because it can go into the matter which will penetrate the metal and get reflected from the next plane and this is the reflected beam and this two part $($ ($)$) different distances this covers longer distance, as I said they are we are familiar with the wave characteristic of X ray these are electromagnetic wave and when this waves interfere waves have been cover different parts they interfere you get an interference pattern or diffraction pattern and you $($) possible to calculate the path difference here, in terms of the D spacing this is the an interplane as spacing D.

Now, path difference is made up of C B this path, you drop a normal from here on to this and a normal from here on to this. So, total path difference is C B plus B D and both of these are equal it is possible to show that both of these are equal say this angle as this angle is also theta. So, this is ninety minus theta this is perpendicular. So, this is theta. So, B D is D sine theta similarly C B also D sine theta. So, total path difference is D sine theta and when this D sine theta is an integral multiple of wave length lamda then the waves are in space you will get in density maximum.

When they are out of phase there will be destructive interference. So, what you will get in certain directions, certain values of theta you will get intense peak whereas, an other values of theta, the intensity of the beam will be minimum lose very less. Now, a little modify you can read like this expression in a different way, say if you say that n lambda over two d is equal to sine theta. We know that, sine theta is always less than one much less than one. So, therefore, you can see in order to get diffraction pattern the wavelength of X ray has to be substantially less than the lattice parameter of the metal, shall I copper I said, the lattice that K alpha radiation the wavelength is 1.54.

Now, if you want to record the diffraction pattern of copper with this copper K alpha radiation it is possible because lattice parameter of copper is around 3.65 remember correctly. So, it is less than much less than. So, around half of the lattice parameter of copper. So, it is possible to record diffraction pattern of copper using copper K alpha radiation

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Now, how do you record the diffracter and this diffraction pattern this is schematically shown over here, say we have a sample which is kept over here this is the incident beam, this is the transmitted beam and the beam is diffracted here and if you go back the the previous one, see here this is this is the angle theta between the diffracted beam and the deflecting beam, but if you extend this this is the transmitted beam if you extend this this is the transmitted beam, then the angle this is theta, this also this is theta you extended this angle is theta angle between the transmitted beam and diffracted beam is twice theta. So, which is shown over here this is the diffracted beam and what you have on this around the circle, we move a count which can be take the intensity of X ray.

So, this so. In fact, the Diffractometer is a device, where you can place this sample on a stage called $($ $($ $)$) meter and this can be rotated, also this can be rotate at about different axis and what you can do, there is a count which moves along this circle and records the intensity as a function of the angle of diffraction that is two theta and which is shown over here, the intensity as a function of two theta and certain places you will find suddenly, you get a very intense peak and this intensity of this peaks will is a function of several factors. It is part from the indices of the plane, it depends on the deflecting plane it depends on the arrangement of grains and the structure and this particular material.

So, in short I have put here, the intensity is a function of structure microstructure and texture how the different grains are oriented. Apart from the crystal planes and the $($ ()) angle that is two theta.

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So, in fact, we can look at it (No audio from 37:35 to 37:43) diffraction after this. Let us, look at Transmission Electron Microscope. Now, we looked at optical microscope and while discussing optical microscope it was mention that resolving power of the optical microscope is of the order of wavelength of let us say wavelength of visible radiation. So, the maximum resolving power that you can get is of the order of two thousand angstrom, but metals are crystalline it is lattice parameters is much large much smaller and therefore, and to record their crystals structure we make use of X ray diffraction takes place.

Now, are there any other better method or where we can resolve the lattice structure much more clearly or. So, one possibility is to $(())$ use much smaller wave length and electrons as we know that if the electrons move at high velocity. It also has a wave character wavelength of X ray is of the order of few angstrom like for copper K alpha radiation it is 1.54 whereas, electrons if they can be accelerated. Let us say for hundred kilovolts or more that wave in its wavelength will be even smaller and it also can penetrate thin foils of metals. In that case, unlike optical microscope.

If we look at this transmitted beam passing through a thin foil of metal and we can magnify with a lens it is possible to resolve much finer structures of metals and this principle of operation of this transmission electron microscope is given in this slide here, unlike optical microscope it rejects an operates in reflection mode, this is operates in a transmission mode.

The sample which is a thin foil is placed over here, you have a filament tungsten filament if it is heated it emits electrons and this electrons can be accelerated by a plain large voltage between the anode and this cathode that is filament a high voltage of the order of hundred, two hundred kilo volts and this and and ray of electrons moving at a very high speed. You know will pass through they can be condensed using lens now unlike in optical microscope we use glass lens. So, here we cannot use glass lens.

Lenses are made up of magnetic coils or electromagnetic operates in the principles of electric and magnetic fields by controlling these, it is possible to make the beam parallel or make and focus it on to the specimen and after it is comes out, you can enlarge it in an objective. In this same way as in an optical microscope and form an image over here, you can also this is the back image plane and you can also magnified it further like eye piece and this magnified image falls on a fluorescent screen and you can see the optically, you can visually, you can see the microstructure of a of metal at a much higher magnification.

You can also put a camera here and record the microstructure, but the sample has to be very thin and which is and it takes a lot of still to prepare the sample and regarding the wavelength it has a direct relationships, the wavelength corresponding to the for the that electron beam can be calculated in terms of using this expression this is root over around one hundred fifty over voltage and this gives you wavelength in angstrom and if you calculate this about even if you use hundred kilo volt you get a wavelength of 0.039 which is much smaller than the wavelength of X rays. So, its resolving power is much more and it can also penetrate thin foil.

So, therefore, it is a $\frac{d}{dt}$ is a resolving power of modern microscopes are very close to this atomic plane resolutions. So, it can go to up the order of angstrom one and two angstrom that is often possible to dissolve crystal plane and transmission electron microscope, but the main problem is sample preparation. This techniques use some of these are listed here, you can take a replica on what you can do on the piece of metal you can deposit a layer of carbon and you can then float that replica by dipping it on water and that replica can be $(())$ onto a copper $(())$ and that grate can be kept or placed in the microscope here as a sample and that carbon foil gives an image of this top surface of the metal and sometime by using some special etching technique, it is possible to extract some of the particles from the metal surface and you can also look at how this particles are distributed can also be seen, but you can get lot of information you can met metallic thin foils.

And this is made by there are two common techniques are electro polishing and iron milling and it needs a lot of skill and practice to prepare a good thin foil for examination under transmission electron microscope.

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Now, what how do you get that like an optical microscope because certain beams are deflected out. So, you get some image contrast in case of an optical microscope. Now, how do you get a similar type of contrast in transmission electron microscope is illustrated over here. So, this is the electron beam passing through the metal foil. The metals are crystalline, the certain parts of the metal then may be favourably oriented to diffract this beam. So, these are the diffracted beams.

The dotted line this is diffracted beam and you have a lens over here, that objective lens here this objective lens will focus these beams is transmitted beams on to this point this diffracted beam on to this point. Now, if you place an aperture over here, then you can block the diffracted beam then you get a $($) this area will appear bright whereas, the areas for were most of this beams are getting diffracted will appear dark. So, the grains and sub grain or sub grains whose orientations satisfies the condition will diffract and they will appear dark. Now, here one may question I said that the sample should be very thin foil thickness should be small enough. So, that the beam does not get observe it just passes through.

Now, this order of foil thickness is a function of several factors like atomic number of the metal the voltage that you applied the kilovolts hundred kilovolt two hundred kilovolt possibly you would be able to examine relatively $(())$ specimen. Similarly, the density of

the foil that the metal of which the foil is made up of is also an important factor and if you are looking the microstructure of aluminium say something like 0.05 to 0.5 micron. You may be able to examine under transmission mode. In case of steel, it has to be much thinner than this and sample preparation of course, as I mentioned needs lot of skill and practice.

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Now, besides transmission electron microscope you also you also use scanning electron microscope. Now, the principles of imaging in a scanning electron microscope is entirely different from conventional microscope. Now, as far as construction is concerned is more or less certain features are very similar to the transmission electron microscope, you will have a filament, you will have an anode the electrons emitted from the filament is accelerate to a high voltage. You will have lens to focus and $($ ($)$) to focus it on the sample.

Now, here in addition you have a magnetic coil here, which can woof this electron beam. Suppose, if this electron beam fall straight here at one point now, we apply some voltage here in that case you can move it from here to here. Similarly, you apply another voltage you shift it from here to here. So, what is done you know there is a scan generator. It generates a scanning voltage and what it does, it scans it shift keep shifting this beam from one point to the another in the form of a like like a something similar to a $T V$ screen and what you collect from here is when this beam strikes a several some of the electrons wave reflected and some penetrate a little.

And some secondary electrons away met at and you get maximum information if you can collect this secondary electron and this collected over here $((\))$ count and this signal is amplified and what is this amplified signal is fed to the oscilloscope and it modulates the intensity of that $\frac{d}{dx}$ the T V screen that $\frac{d}{dx}$ is generated and that is how you generate the image of this surface over here and usually the sample is slightly tilted and. So, basically. So, here you do not have that type of that imaging is entirely dependence on how is well, you can converge the beam to particular point if that point is very small it will have high resolving power and how will you can scan an at what interval you can record that intensity and modulate to generate the image of the surface.

Now, main advantage of this type of microscope is, its resolving power definitely may not be as high as that of transmission electron microscope, but it has one important property that is depth of focus. So, like in optical microscope if you want to see the micro structure at a very high magnification to have to have excellent surface finish it should be $((\cdot))$ finish. You should be able to bring that objective very close to the sample which whereas, in this particular case it is not that critical the sample preparation a sample surface can be much rough even you can record the micros at fractured surface as well.

So, with this what we have looked at to the… We looked at main features of three different types of microscope that we use, that is optical microscope, scanning electron microscope. We also looked at X ray diffraction techniques we look we stated the $(())$ condition under which diffraction takes place that is $((\))$ law.

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 $\begin{bmatrix} \begin{smallmatrix} 0 & \text{GET} \\ 1.1 & T. & \text{KGP} \end{smallmatrix} \end{bmatrix}$ $2d\sin\theta=m\lambda$ $\frac{m\lambda}{2d}$ = Sin θ < 1 $rac{2d}{m}$ $\overline{n\sqrt{\hbar^{2}+}}$ $1, 2, 3$ mh mk, ml

It may be you works for it to look at the Brag law a little more carefully what has been mentioned here that is 2 d sine theta equal to m lamda which is can be written like this and if it is possible to find out what should be the wavelength. So, that you can record diffraction pattern. So, this is the lattice parameter, this is the miller indices of the reflecting plane and this is an integer it calls the order of the diffraction patterns this can $\frac{\partial u}{\partial x}$ have value 1 2 3 etcetera and very often we say in X ray diffraction instead of the saying the reflection comes from plane h k l, we say the reflection comes from n h, n k, n l.

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Now, to record the diffraction pattern one point is very critical this is the incident beam, this is the transmitted beam, this is the diffracted beam and as has been mention its obtains an angle two theta. Now, this is the reflecting beam. Now, this diffraction occurs only if this condition is satisfied. Now, suppose your beam is monochromatic lamda this is fix. So, to get a diffraction pattern at this point for this particular place you have to have a particular crystal which will satisfy this relationship and this can be done in two ways, if you have multiple crystals one of this planes some of particular plane will satisfied of particular crystal will satisfy this which can be visualize like this.

You have a several planes, select this and all planes which are sustaining an angle theta will give you a diffracted beam and if you have a very large number of crystals you can visualize that means, you have diffracted beam coming out in the form of a cone and this type of cases where your sample is made up of large number of crystals. We called this diffraction techniques as a POWDER diffraction technique and in physical metallurgy mostly we will talk will talking about or we will be this is the technique which is most commonly used in physical metallurgy that is also possible to record diffraction pattern of single crystal, but in that case you need to since this becomes fix to satisfy this you must vary this becomes variable.

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And in that case, that type of diffraction pattern technique is called Laue technique and nature of the diffraction pattern will be totally different. In case of a powder diffraction pattern in you get a diffracted beam comes in the form of a cone which is something like this. So, if you put a suppose it is possible to put a film surrounding it, you will get in the film this transmitted beam, you will get diffracted lines like this. So, with this we we come to the end of today's lecture we looked at thermal analysis thermal analysis it is important because it helps you to determine transformation temperatures.

It is also possible to know a little bit of information about the thermodynamics of transformations to some extent. There are other techniques also to follow the transformation like measuring other physical properties like co efficient of linear expansion $(()$ magnetic property if the material is magnetic, but never the less thermal analysis has been most common extensively used to look at the transformation processes in materials. We also looked at the principles of my or how we look prepare samples for microscopic examinations optical microscope the simple polishing and etching.

You can look at you can look at the microstructure in a deflection mode, if you go to that transmission electron microscope. You have to prepare a thin foil. On the other hand, we looked at scanning electron microscope, where sample preparation is not that critical. You can even look at the structure of a fractured or very rough surface with this we finish today's lecture. And in the next lecture, we will talk about mechanical properties. We know am in certain types of mechanical properties which are important, and how they are measured. Thank you.