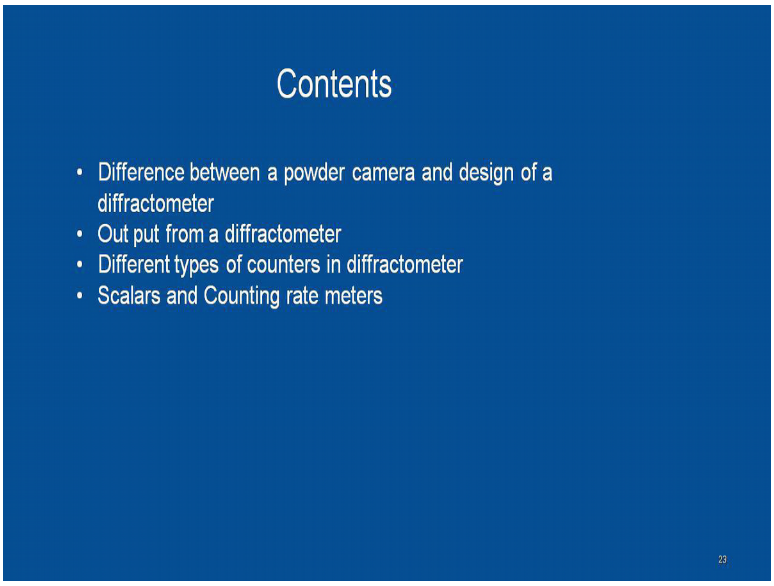
X-Ray Crystallography

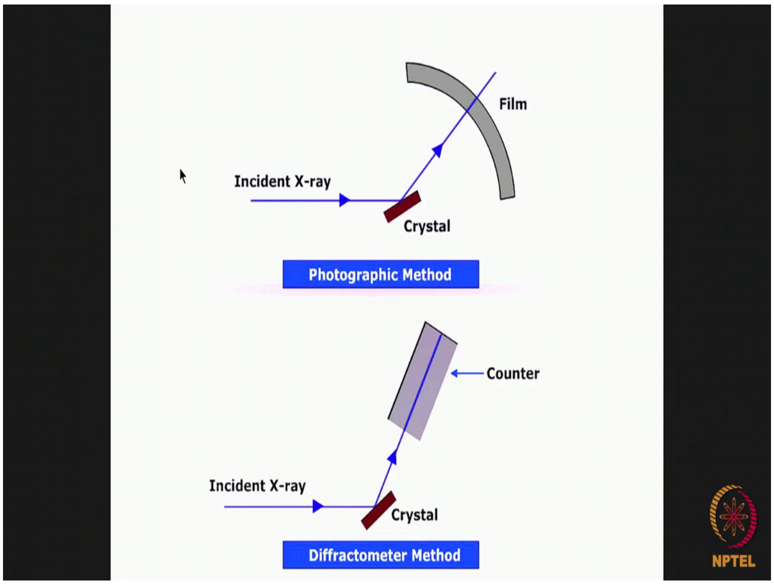
Prof. R. K. Ray
MN Dastur School of Materials Science and Engineering
Indian Institute of Engineering Science and Technology, Shibpur
Department of Metallurgical and Materials Engineering
Indian Institute of Technology, Madras

Lecture - 14
Diffractometer Measurements

(Refer Slide Time: 00:18)



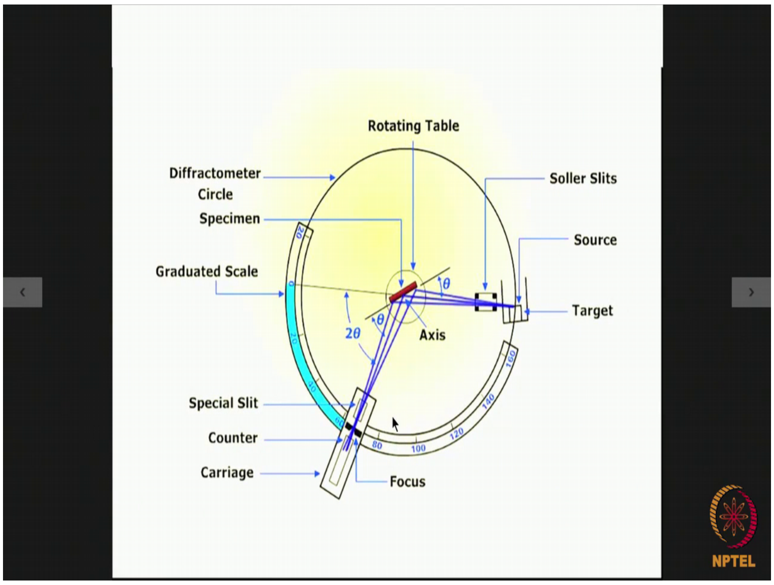
(Refer Slide Time: 00:30)



In this lecture, I shall deal with the method of acquiring X-ray diffraction patterns using a diffractometer. First I will discuss the fundamental concepts and the design of a diffractometer. Then I will talk about the measurement of XRD patterns using counters thirdly I will talk about some simple circuitry that are used for measuring the number of pulse counts by the counters. And finally, I will talk about the kinds of specimen that can be used in a diffractometer. You see up till now I talked about the different types of X-ray cameras for diffraction pattern measurement or according of diffraction patterns.

So, we had the incident X radiation a crystal or a poly-crystal here and the diffracted beams recorded in a photographic film. So, this is the photographic method. So, to say nowadays the diffractometer method has become more popular and most of the X-ray laboratories throughout the world do the diffraction work using a diffractometer this is because the ease of acquiring a diffraction pattern and other relevant advantages. Now in the diffractometer method we do not use the X-ray film to record the diffraction pattern instead we use what is known as a counter. Now this counter is basically a gas counter which I talked about in a previous lecture any way I will come back to it again in this lecture.

(Refer Slide Time: 03:01)



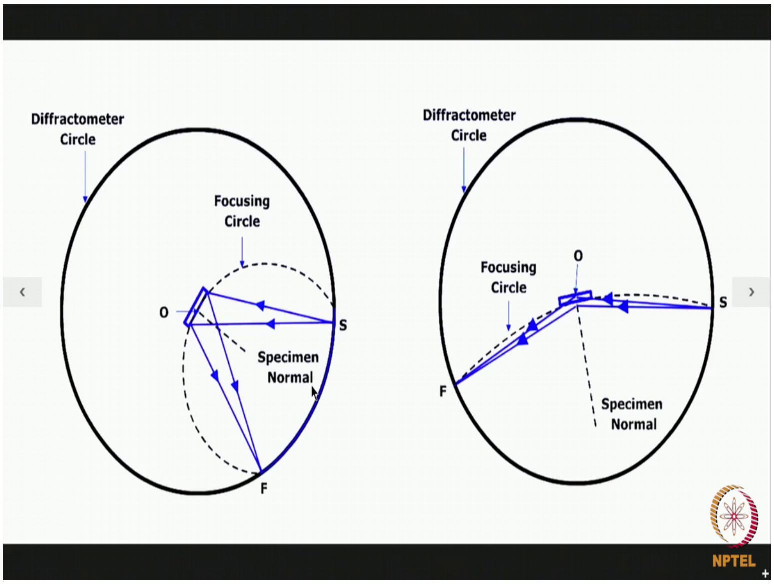
Now what a diffractometer looks like you see this is a cross section of diffractometer equipment. So, this big circle is known as the diffractometer circle at the centre you have a rotating table and where the specimen is kept now this is the source of X-rays and a divergent beam of X-rays are allowed to fall on the specimen and the diffracted beam is recorded in a counter which is mounted on a carriage that can move over a graduated scale.

For example, say if a particular type of HKL planes are diffracting the incident radiation then we can fix the value of theta corresponding to that HKL planes satisfying brag relationship and; obviously, this angle the angle between the incident radiation and the diffracted radiation is 2 theta. So, in the diffractometer what we do we first find out what are the atomic planes from which we would like to have X-ray diffraction taking place or if we do not know suppose from the sample I want to find out the diffracted radiation from all the HKL planes that are present in the poly crystalline sample.

So, what we do we keep on rotating this sample with respective you know this axis starting from a very low value of theta and going up to a high value of theta. So, as we move along there will be planes which will be giving raise to the diffraction patterns and those will be recorded in the counter. So, you see that the specimen and the counter they must be coupled together. So, that when the specimen moves by an angle theta the counter moves by an angle 2 theta. So, that it will be in the correct position to record the diffracted beam.

Now, we normally use a flat specimen in the diffractometer and this is due to the fact that we want to record the diffracted beams from a particular atomic plane focused on to the counter. So, we want to use the focusing action of the diffracted beams. So, that the intensity of the diffracted beam can be increased many times you see the diffracted beams anyway are quiet weak. So, if we can do a focusing of the diffracted beams as we do in a focusing camera in the powder method then that will give us a big dividend.

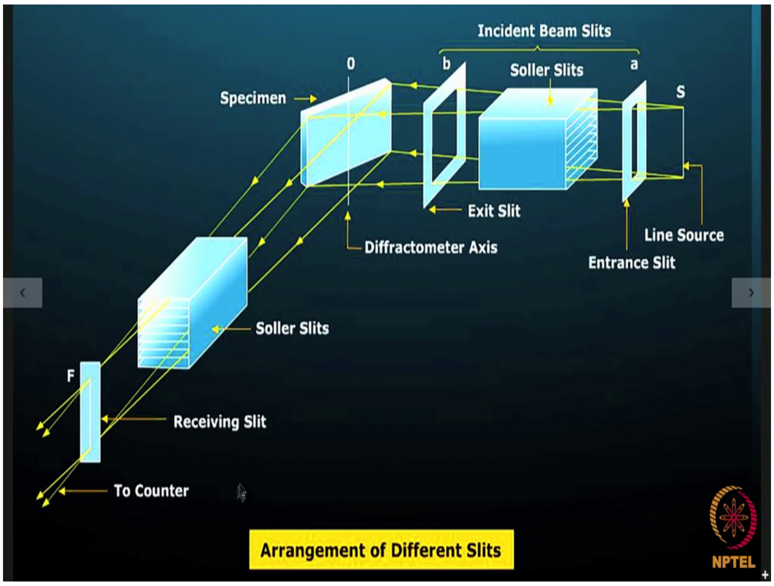
(Refer Slide Time: 06:48)



So, say for example, if this is the diffractometer circle this is the source of X radiation that is falling on the sample and this is where the recording the count this is the position of the counter here which records the diffracted radiation now what we essentially do is we try to keep the sample you know to make you know a tangent on a so called focusing circle like this. So, this size of the focusing circle will depend on the value of theta. So, for a smaller value of theta the focusing circle will be bigger for a higher value of theta it will be smaller and the centre of that focusing circle lies on the specimen normal.

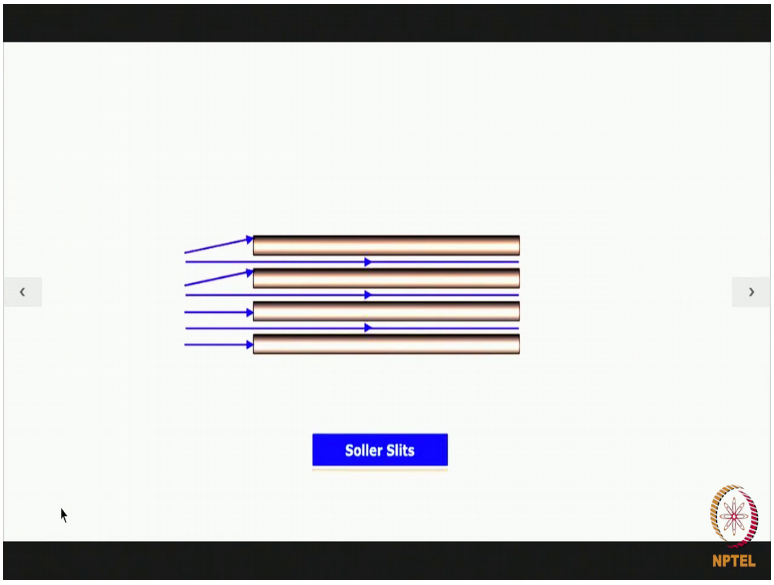
So, this is the arrangement for recording the diffracted patterns the forward direction and this is the kind of arrangement we have for recording in the backward direction. Now there is an elaborate arrangement of slits to control both incident radiation and the diffracted radiation. For example, the source of X-rays that we use is normally a line source.

(Refer Slide Time: 08:20)



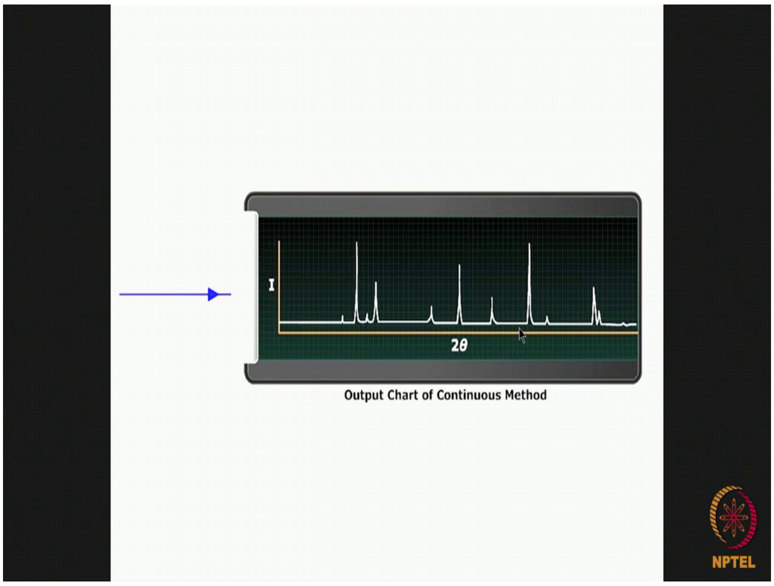
So, if it is a line source then X-rays will be produced along from this line and those will be not only in the plane of the diffractometer circle, but also above or below it. So, in order to keep the focusing geometry intact we must see to it that the divergent beam of X-rays are made parallel to the diffractometer circle plane. So, that is the reason why we use some slits these are called the solar slits I will show how they operate and then we have got 2 exit slits this is their specimen. Then the diffracted radiation again passes through a solar slit this is the receiving slit and it goes to the counter.

(Refer Slide Time: 09:21)



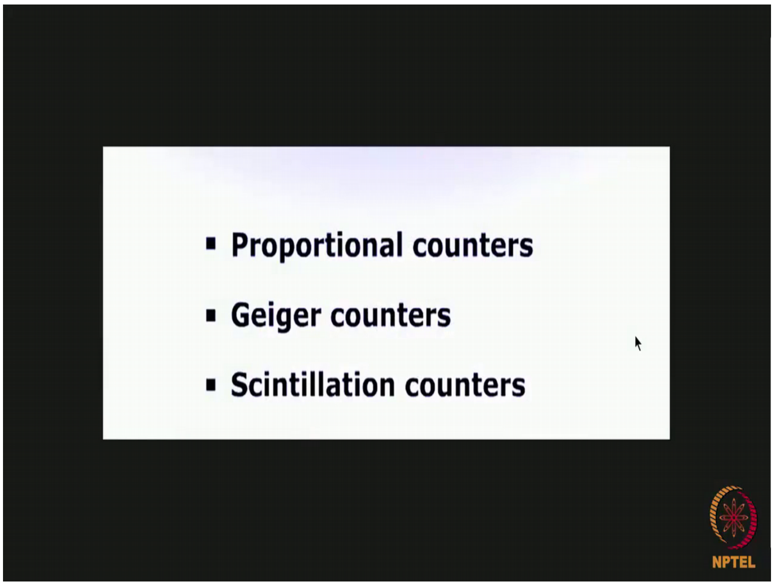
Now what is the function of a solar slit is nothing, but is making up of a large number of flat metallic plates. So, if you have a divergent beam of X radiation it allows only a parallel beam to pass through.

(Refer Slide Time: 09:42)



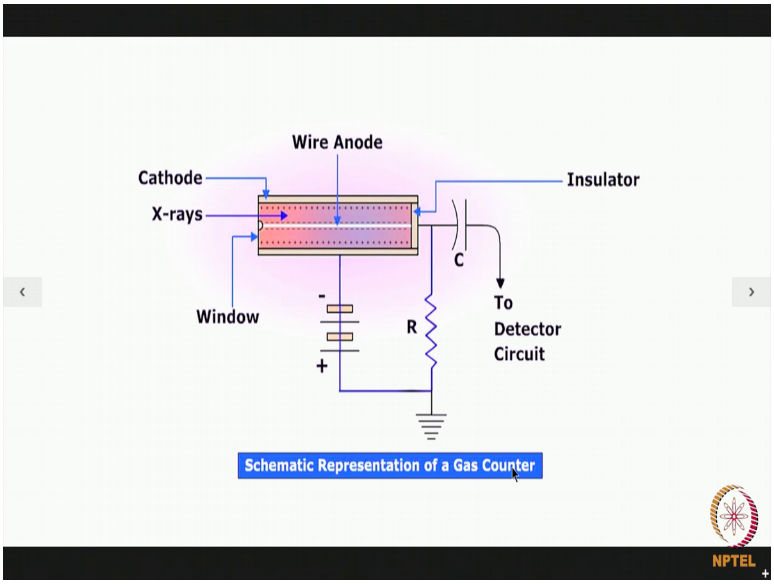
So, this is the function of a solar slit and a output from the diffractometer it looks like this if fact it gives you a direct plot of the intensity versus 2 theta. And these are the diffraction line positions as you can see and what you see here as lines in this pattern these are the same lines which you observe on a photographic film in a Debye Scherer or a focusing camera, but here we have to you do not have to plot the total intensity of the lines versus 2 theta as we have to do in case of the photographic method here we get the output directly as I versus 2 theta plots.

(Refer Slide Time: 10:54)



Now, so far as the counting of the diffracted x radiation is concerned we use three types of counters namely the proportional counters Geiger counters and scintillation counters.

(Refer Slide Time: 11:01)



Now, this is a schematic representation of a basic gas counter with some modifications it can be used as a proportional counter and also as a Geiger counter now the mechanism here is this we have got a cylindrical metallic vessel at the centre of which there is a thin wire anode. So, this vessel is made the cathode and this wire passing through the centre is made the anode X-rays are allowed to the diffracted X-rays are allowed to fall out to enter this vessel through a window over here this is an insulator and this is a resistor that is kept outside the gas counter.

Now as you can see here in a normal gas counter it is filled with a gas and whenever we have a voltage say about 200 volts between the cathode and the anode the gas inside will be ionised. So, the positively charged ions will go towards the cathode and the negatively charged electrons will come closer to the wire anode. And in this way sufficient amount of charge builds up and that can produce a current in the outside resistor.

Now, if we have say a voltage between the cathode and the anode which is of the order of say 600 to 900 volts then what will happen within the gas counter.There will be lot more ionization lot more electrons will be produced and naturally the current that will build up will be lot more than a normal gas counter. Now in a normal gas counter where the voltage between the cathode and the anode is of the order say 200 volts it produces a current in the outside resistor r of the order 10 to the power minus 12 ampere or even less now if we use the gas counter as a proportional counter by applying a high enough voltage between the cathode and the anode say the voltage given is over 600 to 900 volts then the amount of current will be lot more.

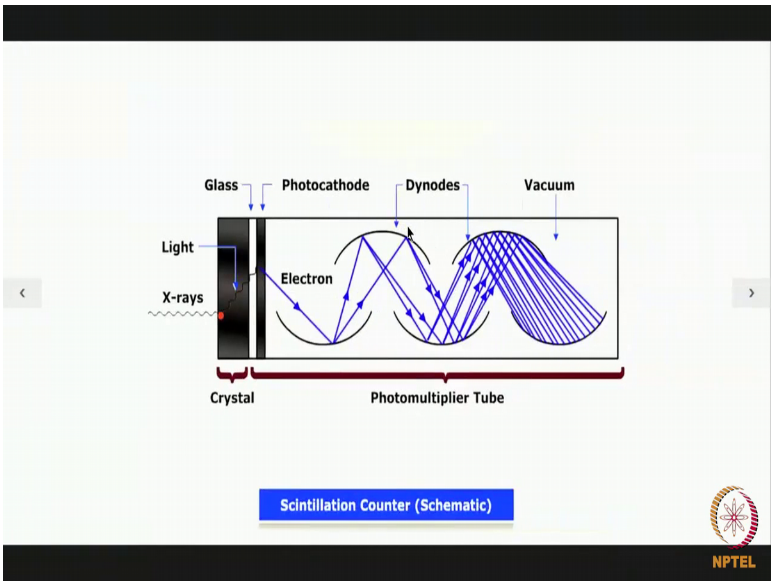
And this current normally can be expressed in terms of the momentary change in the voltage of the wire of the wire anode and that can expressed as several millivolts. So, in a proportional counter the you know the X-ray counter they produce a momentary change in the voltage of the order of several millivolts. Now in the same way if the voltage in a gas counter is increased by several 100 volts they the voltage pulse that is produced is of the order of say 1 to 10 volts and in that case the equipment is known as a Geiger counter.

So, a proportional counter and Geiger counter are basically based on a simple gas counter with a difference that the voltage between the cathode and anode in a proportional counter is much higher than in a normal gas counter and in the Geiger counter it is even higher than that of a proportional counter.

(Refer Slide Time: 15:40)

So, this is a photograph of a portable Geiger counter the other type of counter which is used is known as a scintillation counter.

(Refer Slide Time: 15:46)



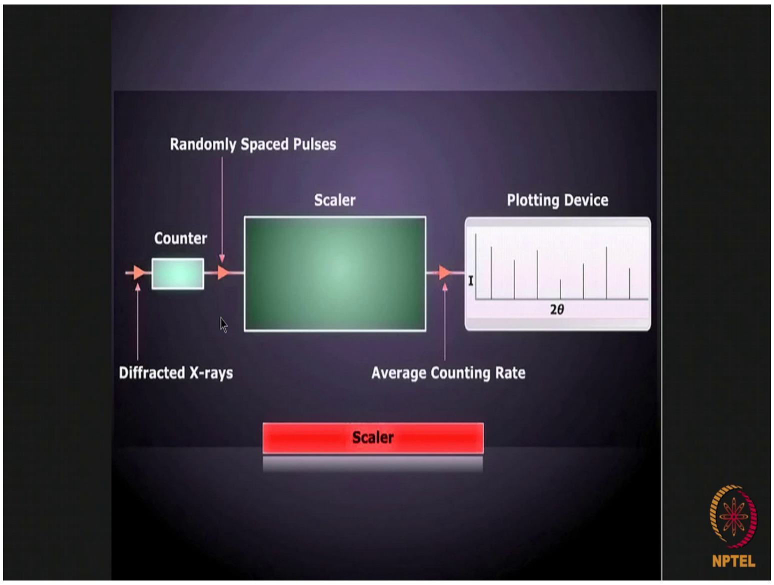
So, what happens in a scintillation counter now as you can see here there is a crystal which is of sodium iodide and it is activated with a little bit of thallium now when X-ray quantum enters this crystal it produces a blue light now this crystal is cemented to a photocathode and a photomultiplier tube. Now the whole apparatus is enclosed in aluminium foil to shield it from outside light. Now the photocathode is made up of an inter metallic compound of caesium and antimony. Now when the blue light enters a photocathode it will produce a number of electrons. Now those electrons are allowed to strike what are known as the dynodes. So, there are metallic dynodes of this type and there are about 10 such metallic dynodes.

So, the electrons striking the metallic dynodes will produce some more electrons and when those electrons will fall on the second dynode they will produce even more electrons and each dynode is kept at a potential of 100 volt plus with respect to the previous dynode. So, the electrons will go on striking against this dynode produce lot more electrons, then those will again come and strike this dynode produce lot more. So, in this way an avalanche of electrons will be produced which will be given by a voltage pulse of the same order as we get in case of a Geiger counter. So, this voltage pulse will be proportional to the X-ray quanta which entered this scintillation counter. So, this is the way these are the three types of counters that normally are used for counting the X-ray quanta that comes out of the that is produced by the diffracted beam.

Now, when it comes to counting of pulses in a counter you see normally the; what we do we measure the average number of counts of the pulses of the electrical pulses produced in a counter by dividing the number of pulses produced within a certain amount of time. So, number of pulses produced divided by the time taken than is taken as an average count. Now if we use a normal mechanical counter where the counting rate is low they can count the counting rate very very accurately, but if the rate of production of electric pulses is rather high then mechanical counters are of no use in that case a we use a scalar now what is the function of a scalar.

Now the function of a scalar is it counts it is capable of counting very high rate of pulse production and in this case for a particular diffraction pattern for a particular you know a particular diffracted intensity coming from a particular series of planes, we can count the average count rate, we can measure the average count rate and that is displayed through a plotting devise on an output which looks this.

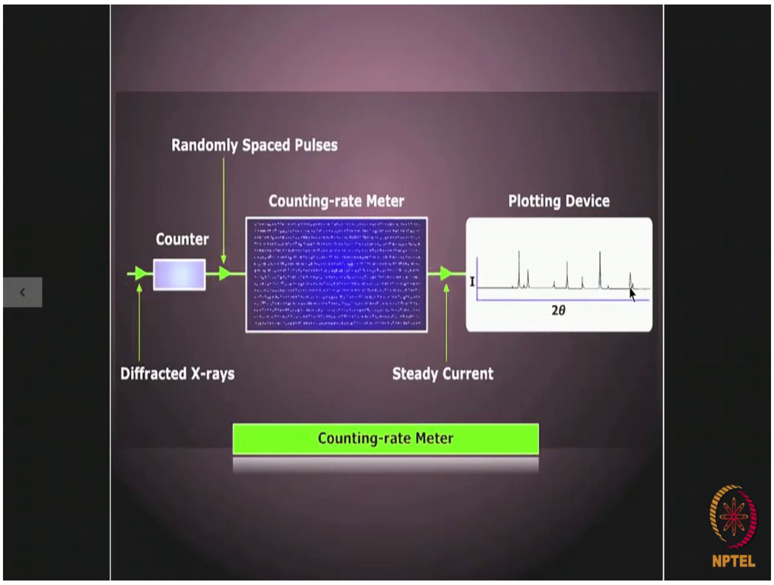
(Refer Slide Time: 20:14)



So, this is these are indicative of the diffracted intensity from different atomic planes in the material. And as you can see here this is an intermittent method at each diffraction line for as for any position of the material in the diffractometer sufficient time is given to find out a sufficient number of counts, and then it is divided by the time taken and you can get an average count rate.

So, this is how it is an intermittent method of producing the X-ray diffraction pattern using a scalar the other circuitry which we used is known as the counting rate meter.

(Refer Slide Time: 21:18)



So, what happens in a counting rate meter it smoothens out the succession of electric pulses that are produced in the counter. So, the diffracted X-rays enter the counter goes to the counting rate meter. So, it smoothens out the succession of the electric pulses and produces a continuous current then that continuous current is plotted in a plotting device as I versus 2 theta plot. So, you see that in this case it is a continuous plot of the intensity versus 2 theta where as when we use scalar it is an intermittent method of getting the I versus 2 theta values in a diffractometer.