


**Characterization of Construction Materials**  
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**Lecture 12**  
**X-Ray Diffraction**

Hello everyone, in this module we will learn about X-ray diffraction which is used quite commonly to understand the construction materials. So, the goal of this lecture will be to just know about the X-rays and how we can use these X-rays to understand the material.


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**X-Rays**



- X-rays are electromagnetic radiation of same nature as light but of very much shorter wavelength.
- The unit of measurement in the X-ray region is the angstrom (Å).
- X-rays used in diffraction have wavelengths lying in the range of 0.5-2.5 Å, whereas the wavelength of visible light is of order of 6000 Å.

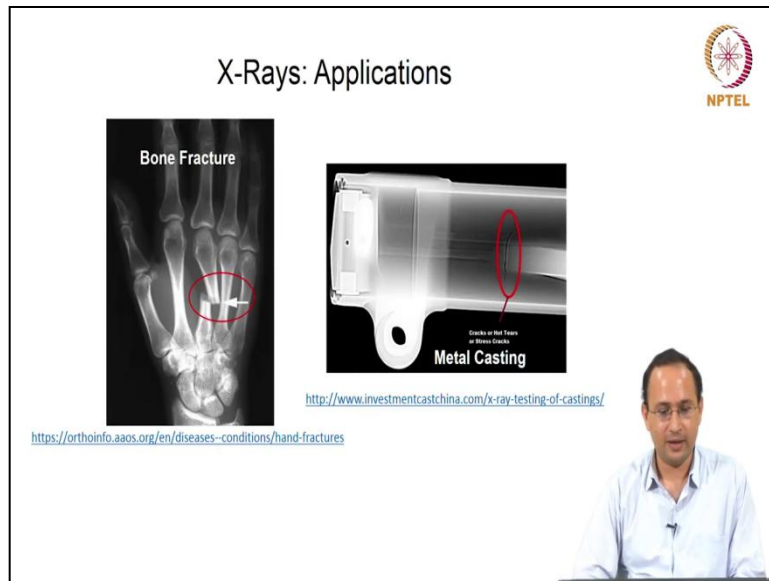
$1 \text{ \AA} = 10^{-10} \text{ m}$



So, we will start with the background. That is very important to know the history. So, X-rays were discovered by German physicist Röntgen in 1895 and he received the first Nobel Prize of physics for this. So, unlike ordinary light X-rays are invisible. You cannot see it and X-rays have more penetrating energy than light and could easily pass through the human body, wood, quite thick pieces of metal and other opaque objects.

These were the observations. So, X-rays were immediately put to use for detecting, for an example, a broken bone or the position of a crack in a metal casting which you will see in the next slide.

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So, here you see these radiographs, you can identify the bone fracture. So, and also similarly, in the casting if there is a crack, you can see it. So, that was the significance of this discovery that without even knowing much about X-rays, they could use these X-rays to identify the bone fracture or cracks when you do casting of metals. Why X-rays? Because they did not exactly know the nature of these rays, so, they said X-rays.

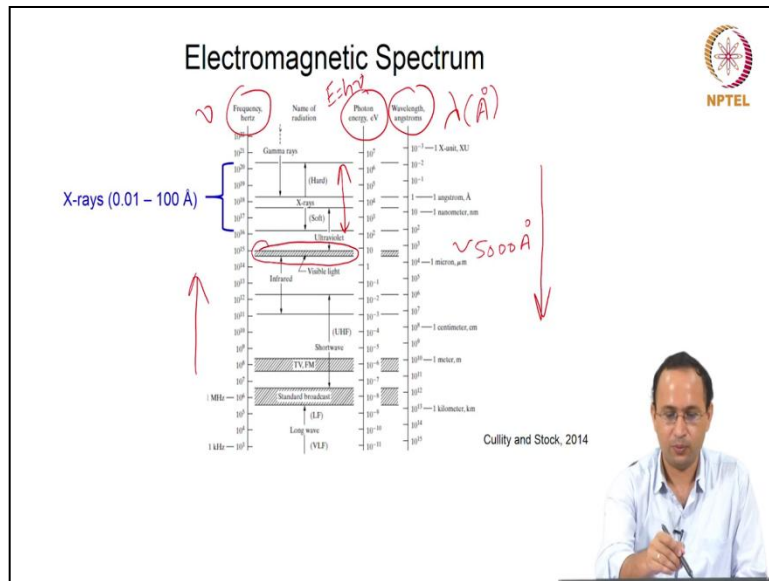
So, that was a huge benefit of these X-rays. So, they were put to use immediately for these applications. So, basically X-rays are electromagnetic radiation of same nature as light, but of very much shorter wavelength. If you look at the electromagnetic spectrum, what you find is the wavelength of X-rays is much shorter than the wavelength of light.

And the unit of measurement in the X-ray region is in angstrom ( $\text{\AA}$ ).

$$1\text{\AA} = 10^{-10} \text{ metres}$$

X-rays used in diffraction have wavelengths lying in the range of 0.5 to 2.5  $\text{\AA}$ , whereas the wavelength of visible light is of order of 6000  $\text{\AA}$ . See the difference? So, X-rays which we use for diffraction are of much shorter wavelength.

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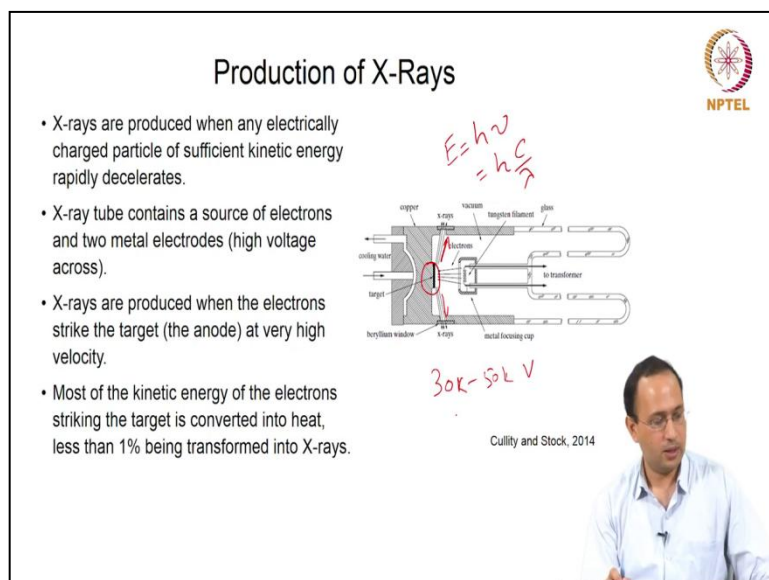
So, here is the electromagnetic spectrum. Here is the visible light, in terms of wavelength in Angstrom it's around 5000 Å, roughly around that range 5000-6000Å and X-rays can range from 0.01 Å to 100 Å. So, in this direction (downward), wavelength is increasing.

When the wavelength is increasing, the frequency will decrease; so, in this direction (upward) frequency is increasing. All of you know the relation between wavelength and frequency. So, you can see where it is. So, it has shorter wavelength than visible light. And this is the range 0.01 Å to 100 Å. Also you can see energy. So, frequency is 'ν' and we know that energy can be expressed in terms of

$$E = h \cdot \nu$$

So, you can say energy increases in this direction (downward).

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So, we know X-rays are invisible, have shorter wavelength that means higher energy.

$$E = h \cdot \nu \text{ (or) } E = h \cdot \frac{c}{\lambda}$$

Also you should remember that shorter wavelength means higher energy. So, now the question comes, how do we produce these X-Rays? If they are so advantageous, it is good to know how they are produced. So, X-rays are produced when an electrically charged particle of sufficient kinetic energy rapidly decelerates. That charged particle could be electron.

So, in this case you see X-ray tube. What does an X-ray tube contain? A source of electron and also two metal electrodes; two metal electrodes should be connected or there should be very high potential which can accelerate these electrons. We are talking about 30kV – 50kV, voltage difference, which is very high, high voltage across these metal electrodes cathode and anode.

And X-rays are produced when the electrons strike the target, which is anode at very high velocity. So you are generating electrons and there is a very high voltage, so electrons are going to hit the target and then X-rays are produced when these electrons strike the target. So, what happens is, most of the kinetic energy (electrons have very high kinetic energy) is converted into heat. Less than one percent is being transformed into X-rays. So, that is how you generate X-rays.


This is the schematic of an X-ray tube (See Figure in slide). You see tungsten filament, here in this case, which you will use to generate electrons connected through transformer. And there is a high voltage across anode and cathode. You see the target metal, the electron hits the target metal and X-rays are emitted and you can get it. These are the directions in this case (upward and downward).

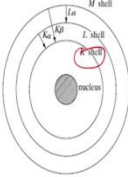
Also since the target will get really hot, you need a mechanism to cool it down. So, you see cooling water that is used to cool the target. This is the typical schematic of X-ray tube.

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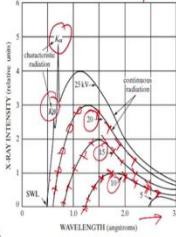
## Characteristic Spectrum

- When the voltage on an X-ray tube is raised **above a certain critical value**, characteristic of the target metal, sharp intensity maxima appear at certain wavelengths.
- The associated wavelengths are characteristic of the target metal and are called **characteristic lines**.
- These lines fall into several sets, referred to as *K, L, M*, etc. in the order of increasing wavelength, all the lines forming the **characteristic spectrum** of the metal.






Cullity and Stock, 2014



X-ray spectrum of molybdenum



So far we know that electrons of very high energy striking a metal target will give rise to X-rays. But we want to probe further. So now, we are analyzing the X-rays which are coming out. They contain various wavelengths and associated intensity.

So you can plot it. Suppose you analyze the X-ray coming out of the target, it will have for an example, in this case, suppose, we are operating at 10 kV that is the voltage difference. So, you will get this mixture of wavelengths and also X-ray intensity changes. First of all, you do not see any X-ray, there is a shorter wavelength, and there are no X-rays in this range.

Then, it starts here, and then you have these different wavelengths of different intensity. Also another way of looking at it, as wavelength is increasing, energy is decreasing. Always remember this relationship.

$$E = h \cdot \frac{c}{\lambda}$$

The wavelength is decreasing that means energy is increasing; wavelength is increasing means energy is decreasing. So, this is called continuous spectrum; that is what is in that X-ray typically.

So, again now you increase this 10 kV to 15 kV, what happens, the curve shifts towards left, and then you see again, mixture of wavelengths having different intensities. The curve is similar, the only thing is that it has shifted towards left, because we have more energy. See, you are supplying more energy which means lambda will be smaller.

You can again refer to this relationship  $E = h \cdot \frac{c}{\lambda}$ . So, if you have more energy means lambda is shorter. So, as you are increasing this voltage, that means you are inducing, you're accelerating the electrons. So, in 20 kV, again you see it shift towards left, you see a similar spectrum. It is called continuous spectrum. It is continuous or white radiation.

But if you see what happens, when the voltage on an X-ray tube is raised above a certain value, in this case, 25 kV, what is happening? Then, the characteristic of the target metal, sharp intensity maxima appear which is not present in other continuous spectrum. Only in this case of 25 kV, you see the appearance of these sharp maxima. So, the associated wavelengths they occur at are called characteristic lines.

And it is very specific to a particular metal. It is characteristic of the target metal. If you change the metal, for an example, this wavelength will change. But what is interesting is like, if you increase it further to 25 to 35 kV, it is not plotted here, what you will see obviously is the intensity will increase but the position will stay same. The, the  $K_{\alpha}$  position will be same( $\lambda$ ). So, it is very unique of a metal, in this case, target metal. It is called characteristic, it is unique.

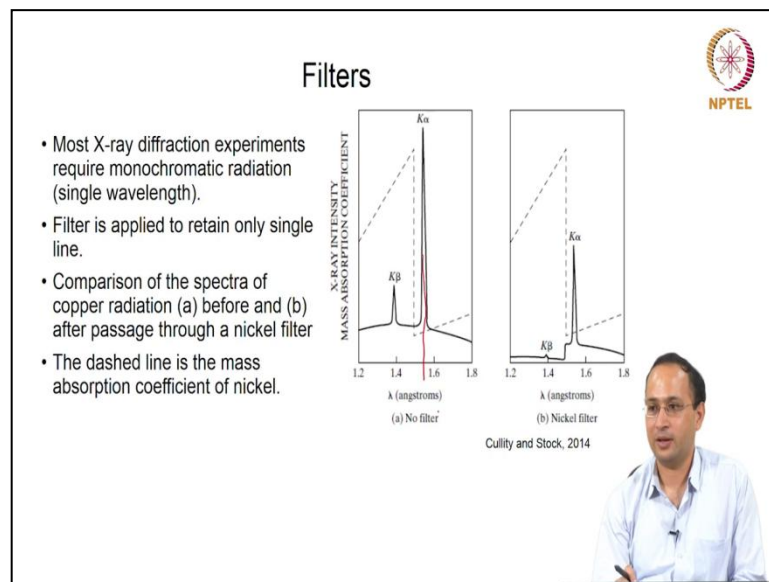
Next question is what gives rise to these intensity maxima, suddenly the intensity is increasing. Basically, all of you have studied this in elementary chemistry. So you have this nucleus, surrounded by different shells – K, L, M, and N. So, you have these electrons moving at really high speed striking the metal.

So, if that electron has enough energy to knock an electron out of this K shell, what will happen, there is a vacancy. Now, the electron from L shell will try to take its place. So, this gives rise to  $K_{\alpha}$ . Suppose there is an electron in M shell, if it tries to take place of the K shell, it is called  $K_{\beta}$ . More likely you will have  $K_{\alpha}$  because you are closer to the nucleus. So, this is the reason why we get these.

Also, you will see  $L_{\alpha}$  because if something can strike and knock out an electron from K shell obviously, it will also remove electron from L shell and M shell. But they will be of much higher wavelengths. So, this is the unique thing where we can use this X-rays to probe, because first of all, lot of experiments like X-ray diffraction, in diffraction experiments we use monochromatic radiation.

Monochromatic means we want only one lambda. Here you are getting that unique lambda. Now you can see  $K_{\alpha}$ ,  $K_{\beta}$ , you have two wavelengths. But there are ways to filter it out. But at least you know that if you have to get a unique wavelength, you can get it. That is the characteristic line of that particular target matter.

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Now, the question comes, lot of experiments we need monochromatic radiation, single wavelength, how do we do it? We can use some filters which will absorb. So, in this case, we see the Nickel filter. So, when you apply a nickel filter, it will have that absorption band between  $K_{\alpha}$  and  $K_{\beta}$ . So, basically it will be able to absorb. So, after applying the filter, you only see  $K_{\alpha}$ , not  $K_{\beta}$ . So, this is one of the ways to filter it out.


Now, you know what gives rise to these characteristic lines. And they are very useful, because you have now a single wavelength. And in this case, what is the wavelength? Very small, you are talking about maybe 1.4 - 1.5 Å. So, these have very short wavelengths, typically that is the order of atomic spacing in crystals, so that is what makes these X-rays useful.

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### X-Rays: Radiography and Diffraction

  
NPTEL

- Radiography was initiated without any precise understanding of the radiation used.
  
- It was not until 1912 that the exact nature of X-rays was established.
  
- This year the phenomenon of X-ray diffraction by crystals was discovered.
  
- This discovery also proved the wave nature of X-rays and provided a new method for investigating the fine structure of matter.




So, diffraction came quite late. So, but, people knew that these X-rays have very high energy; they can probe thick pieces of metal, wood. So, radiography was initiated without any precise understanding of the radiation used. You can get a radiograph, you place something before X-rays and you will get a radiograph. It was not until 1912 that the exact nature of X-rays was established. It took quite a bit of time, although the X-rays were discovered in 1895 by Röntgen.


This year (1912), the phenomenon of X-ray diffraction by crystals was discovered. So this discovery also proved the wave nature of X-rays and provided a new method for investigating the fine structure of matter.

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We will now consider the geometry and structure of crystals in order to discover what there is about crystals in general that enables them to diffract X-rays.




So, now we know what X-rays are, how they are generated, what is so unique about X-rays, which we can use to probe the material, crystalline materials. So, now we will now consider the geometry and structure of crystals, in order to discover what there is about the crystals in general, that enables them to diffract X-rays. So, now you know about the X-rays. We know that X-rays can be used to probe crystalline materials.


So, for that we need to understand what are the crystals? So, now I will focus on crystals basics. So, then we can connect X-rays to crystals. That is the goal.

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### Crystals



- Crystalline material: A solid with atoms arranged in a repeating or periodic array over large atomic distances
- Long range order exists
- Repetitive 3D pattern
- Unit cell – Basic repeating unit
- Directions – Vectors in 3D space
- Planes – Surfaces in 3D space defining layout of atoms
- Properties depend on the spatial atomic arrangement



So, crystals, crystalline material is a solid with atoms arranged in a repeating or periodic array over large atomic distances. There is long-range order; there is a periodic arrangement, long-range. A repetitive 3D pattern, you notice the repetitive 3D pattern. In all three dimensions,

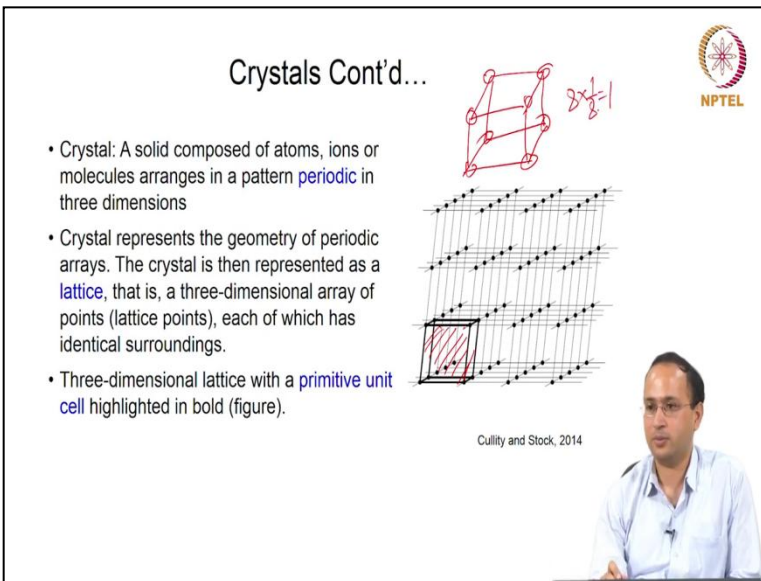
there is a repetition. And the Unit cell is a basic repeating unit. That is the cell which gets repeated. And the Directions in the crystals are the vectors in 3D space.

And the Planes are surfaces in 3D space defining layout of atoms. These are just basic terminologies and obviously the properties of crystal will depend on the spatial atomic arrangement; where they are placed.

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Crystals Cont'd...

- Crystal: A solid composed of atoms, ions or molecules arranged in a pattern **periodic** in three dimensions
- Crystal represents the geometry of periodic arrays. The crystal is then represented as a **lattice**, that is, a three-dimensional array of points (lattice points), each of which has identical surroundings.
- Three-dimensional lattice with a **primitive unit cell** highlighted in bold (figure).




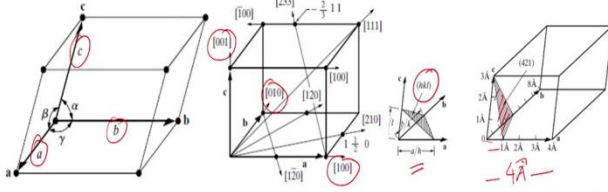
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So, crystal is a solid composed of atoms, ions or molecules arranged in a pattern periodic in three dimensions. From this picture, you can see its periodicity. And crystal represents the geometry of periodic arrays. The crystal is then represented as a lattice, that is, a three-dimensional array of points, each of which has identical surroundings, you can see, this is the lattice (See figure). It has identical surroundings and this is the thing which gets repeated and three-dimensional lattice with a **primitive unit cell** highlighted in **bold (See figure)**. It is called primitive because it has one atom per unit. One atom is because - this is the primitive, so it has eight atoms and each atom is shared by eight unit cells. So, you have  $8 \cdot (1/8) = 1$ .

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### Points, Lines, and Planes





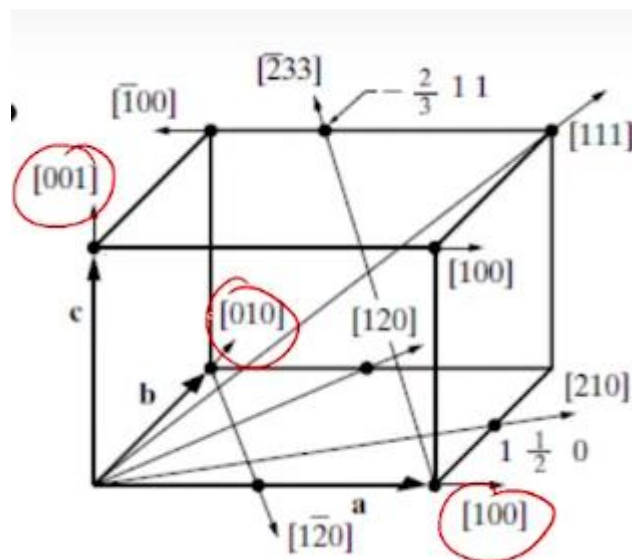
Miller Indices: the reciprocals of the fractional intercepts which the plane makes with the crystallographic axes.

Axial lengths	4Å	8Å	3Å
Intercept lengths	1Å	4Å	3Å
Fractional intercepts	1/4	1/2	1/3
Miller indices	4	2	1

Cullity and Stock, 2014

Little bit of background. You may already be familiar with this. But just for completion it is important to review the basics. So points, lines and planes. In crystal, specifically, we are talking about a, b, c - the lines in three dimensions along x, y, z. Also what is important is the angle between these.  $\gamma$  is angle between a and b;  $\alpha$  is between b and c;  $\beta$  is between a and c.

So these are six things we need to know, if you want to figure out which crystal system it belongs to. Lines – (Refer Figure 1)

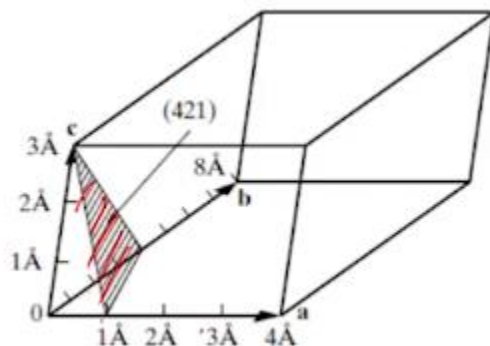


**Figure 1**

As you see here, if you have to represent this is [001], this is [100] in this direction, [010] this is the vector.

Now how do we define planes? So for that we will use the Miller indices. That is the system Miller developed to define a plane.

So in this case, suppose we have this plane which cuts 'a' axis here, 'b' axis here, 'c' axis here. This is the plane we are talking about (Refer Figure 2).



**Figure 2**

Basically, it cuts 'a' axis at 1 Å, 'b' axis at 4 Å, 'c' axis at 3 Å.

Total length is 4 Å ('a' axis), this is 8 Å ('b' axis). That is the total length clear. Now, we know the axial lengths 4 Å, 8 Å, 3 Å.

Intercept length - This plane is cutting these three axes at different points. So intercept with 'a' is 1 Å, intercept with b is 4 Å, intercept with c is 3 Å. Now, we calculate the fractional intercepts so it will be (1/4), (4/8), (3/3), so you get (1/4), (1/2), (1). Now you have to get rid of these fractions, so you have to invert it. Fraction is (1/4), (1/2), (1), now you invert it, so becomes 4, 2, 1. So, the Miller indices will be (421).

Why this inversion, you must be wondering why we have to invert? Sometimes what happens, the plane does not cut an axis. So, you may say the intercept is infinite. So, rather than dealing with infinity, it is better to deal with zeroes. How do you get 0 from infinity? You divide.

$$\frac{1}{\infty} = 0$$

That is why and that is how this system was developed.

So, basically what are the Miller indices? It is the reciprocals of the fractional intercepts which the plane makes with the crystallographic axis. Any plane will make some intercepts. So, you have to calculate those intercepts and then it is a reciprocal of the fractional. That is the key-reciprocals of the fractional intercepts. So, in this case, this plane will be (421).

So, any plane with these indices – (hkl) (Refer Figure 3) will have (a/h) intercept with a, (b/k) intercept with b and (c/l) intercept with c.

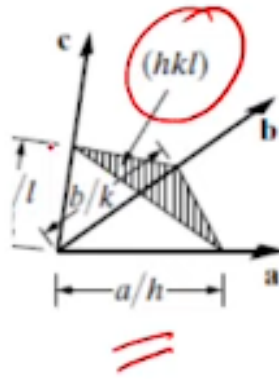


Figure 3

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Miller Indices Cont'd

$(100)$   
 $1 \infty \infty$

(100)

$(200)$

(200)

$(110)$

(110)

$(\bar{1}10)$

(\bar{1}10)

$(1\bar{1}1)$

(1\bar{1}1)

$(102)$

(102)

Observation: The lines of lowest indices have the greatest spacing density of lattice points.

Cullity and Stock, 2014

Again, going over Miller indices again, these are the planes so you have a,b,c - three crystallographic axes. So, plane (100) is this plane. It will have intercept of 1, only with axis a, and the others are 0, because this plane is having intercept of 1 with 'a', infinity with 'b', as it is not cutting 'b'.

What is the reciprocal? (100) and the way you represent these planes is in parentheses - that is how you designate a plane. So, similarly now (200) will be somewhere between. How do you get this (200)? Now, suppose your intercept is 1 here, in this case your intercept is (1/2) and again y-intercept with 'b' is infinity, intercept with 'c' is infinity, now, you reciprocate it. It becomes (200).

Now you can work it out. The idea is you have to find the intercepts with three crystallographic axes. And then, get the reciprocal. That is how you define the plane. And when there is a

negative sign then, you put a bar. So, you see here this plane represented as  $\bar{1}10$ . So, you can practice and see how you get these Miller indices.

On your right, what you see so, again these are the planes. Lines, for an example, we are now not focusing on c axis. We are only talking about 'a' and 'b' - two dimensions. So, this is your (10) line. This is (13),  $(4\bar{1})$ . What do you notice? See these planes, you can again get it. You can calculate and arrive at these Miller indices. So, observation is the lines of lowest indices have the greatest spacing density of lattice points.

You see (10) you have more spacing, as you go towards higher indices,  $(4\bar{1})$  your spacing is reducing. But these are general. So, the lines of the lowest indices, lowest is like (10) have the greatest spacing density. That tells you about the arrangement of atoms if you are talking about the planes. So, it gives you an insight. If you compare two different planes, it will tell you, where you have closer spacing.