

**Failure Analysis & Prevention**  
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**Lecture – 32**

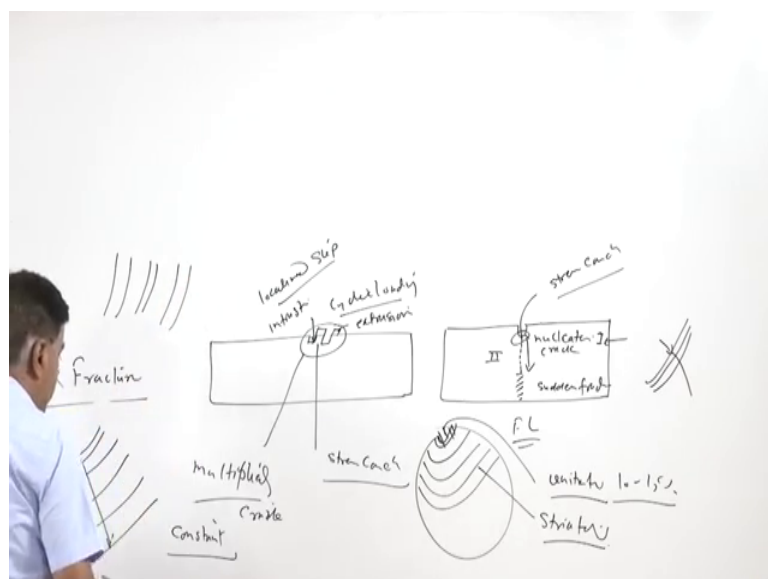
**General Procedure of Failure Analysis: Determination of Type of Fracture III and Chemical Analysis**

Hello, I welcome you all in this presentation related with the subject failure analysis and prevention. And nowadays we are talking about the general procedure of the failure analysis and under that we have talked about so, many aspects like collection of the background information, preliminary examination of the failed component, non destructive testing of the field component and destructive testing microscopy, microscopy of the fracture surfaces and then metallographic aspects of the field component and thereafter we also talked about the determination of the type of fracture.

Under that we have talked about the dimple fracture cleavage how to determine the that the way by which fracture has taken place, considering the microstructure microscopy and micrography of the fracture surface.

So, whether it is ductile fracture having the dimple, dimples on the fracture surface or it is brittle with the inter granular fracture or a cleavage facets or any other form of the fracture unlike hydrogen induced cracking or the creep.

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Similarly, now there is one more fracture; which about which we need to talk is the fatigue fracture. This is related with the basically the determination of the fracture only. So, here we know that even the fatigue can occur not just on the components with the stress regions like this, but also the components which are very smooth.

So, then a component with the stress regions or unique features, which will be localizing the residual or localizing the applied stresses; So, they will be basically increasing the stress concentration in those areas and thereby facilitating the nucleation of the crack easily nucleation of the crack easily. Then once the crack is nucleated it will be growing sequentially until the sudden fracture takes place. So, the nucleation of the crack is a stage 1, the growth of the crack is this stage 2 and the sudden fracture is the stage 3. So, and each one will have the different kind of the surface morphology.

So, in order to understand and appreciate; So, the all these stages you see if there is a notch then the nucleation stage is completed earlier because of the localization of the stresses, but thereafter the material properties will be determining the rate at which it will be growing.

On the other hand when the component is new even then also under the cyclic load conditions, under the cyclic load conditions due to the very localized the slip which is taking place at the micro level at the specially at the surface. So, it will be leading to the creation of the intrusions and extrusion at the surface and these intrusions and extrusions of after a sufficient number of the cyclic loading, it leads to the generation of the irregularities at the surface like this.

So, one which is below the surface will which is termed as intrusions and other one is termed as extrusion. So, in any case these will be creating the irregularities at the surface, and thereby they will be causing the increase in the stress concentration.

So, in this particular case since it takes some time to create to nucleate a crack like discontinuity, well in the case when there is already notch which is acting as a stress raiser. So, the life fatigue life in this case will be somewhat lower as compared to the case, when there is no a stress raiser and this smooth streamline smooth finished component exist.

So, basically; So, this is one aspect that the surf crack nucleation is one stage and whenever such kind of things happen, we will find that multiplicity of the crack is present multiple cracks are present a near the fracture initiation site; while in this case there may be a sharp crack sharp zone where from it has been initiated and subsequently it has a grown.

So, if we see these cross sections, which have failed in fatigue manner. So, the location where such kind of nucleation has taken place that will be showing the different kind of the morphology, and that is termed as the initiation zone. It takes life of about 10 to 15 percent depending upon the surface roughness or the kind of a stress raisers, which are present on the fracture on the component. Thereafter, such kind of the beach marks and at micro level these are termed as striations are formed on the surface.

So, the gap between the individual striations corresponds to the growth of crack, roughly in each loading cycle. So, the spacing is also used to characterize the rate at which the crack is growing. So, if the gap between these striations is large will be indicating that crack growth rate was so high as compared to the case when the gap between these striations is low.

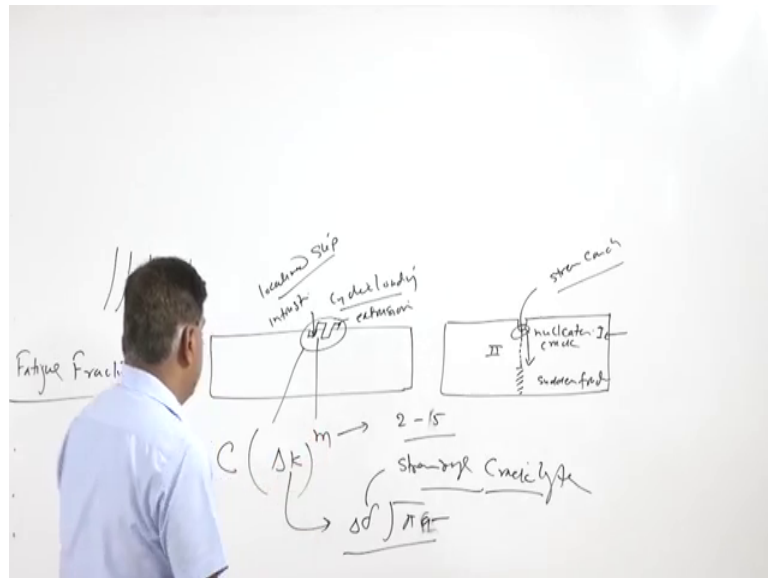
Variation in crack grow; So, there can be a continuous change in spacing between the striations, in general the space this gap keeps on increasing with the increase of the crack size. So, initially the growth rate is less and then this spacing will keep on increasing, because of the increasing the stress amplitude actual stress amplitude or the stress intensity factor  $K_{Ic}$ .

So, here the spacing will keep on increasing, this happens even in case when the load is the load fluctuation or the stress amplitude is constant.

But this can also happen when the load is fluctuating when the maximum magnitude of the tensile load especially is wearing significantly, in that case we will find lot of variation in the spacing between the striation. So, the in over a narrow zone if the variation in the spacing between the striations is large, that can indicate the a possibility of the a fluctuating load conditions instead of instead of the constant, constant stress amplitude.

Now, the efforts have been made to study that how the crack grows especially during the second stage so, that the life of the component under the fatigue conditions can be estimated, can be determined.

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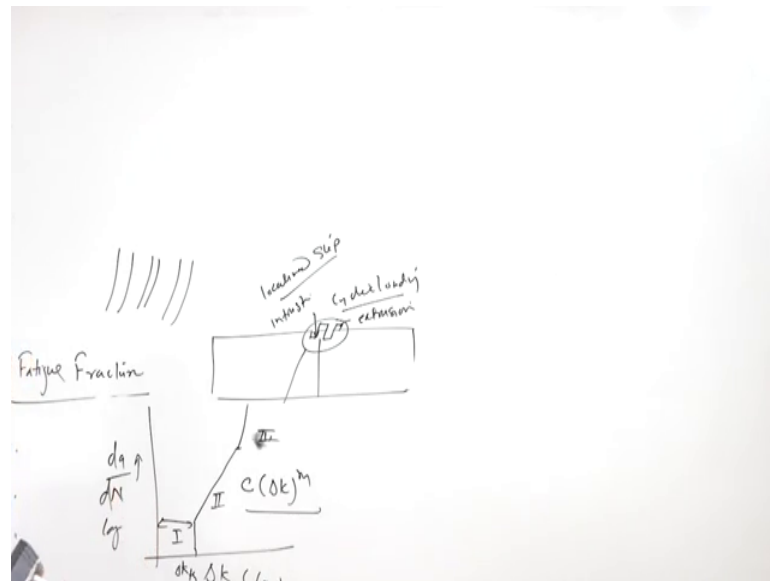


So, the  $da$  by  $dN$  is the typical formula, which is used to indicate  $C$  is the constant and  $\Delta K$  is the stress intensity factor range  $m$ ;  $m$  is the constant.

So, depending upon the material system value can vary from like 2 to 3 or even more it can vary go up to the 5 also and the stress intensity factor range is basically the  $\Delta \sigma$  means the kind of stress variation, which is taking place the stress range under root  $\pi C$  or. So, here we can write like they  $a$  is the crack length, crack length and the  $\sigma$  is the stress range basically the  $\sigma \Delta \sigma$  is the stress range.

So, as the crack size grows in the  $\Delta K$  will be increasing even for the given stress range and the rest of the things are other constants. So, this will indicate the rate this will indicate that with the increase of the crack size for a given stress range also there will be increase in the growth rate of the crack, which we can show with the help of this very commonly used figure noise] where in  $x$  axis we plot  $\Delta K$  and  $da$  by  $dN$  in the  $y$  axis.

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So, this indicates the rate of the crack growth especially in the second stage.

So, minimum variation in the stress range is required for minimum the stress intensity factor range is needed for growth of the crack they are after, the before that their crack will be non-propagating type.

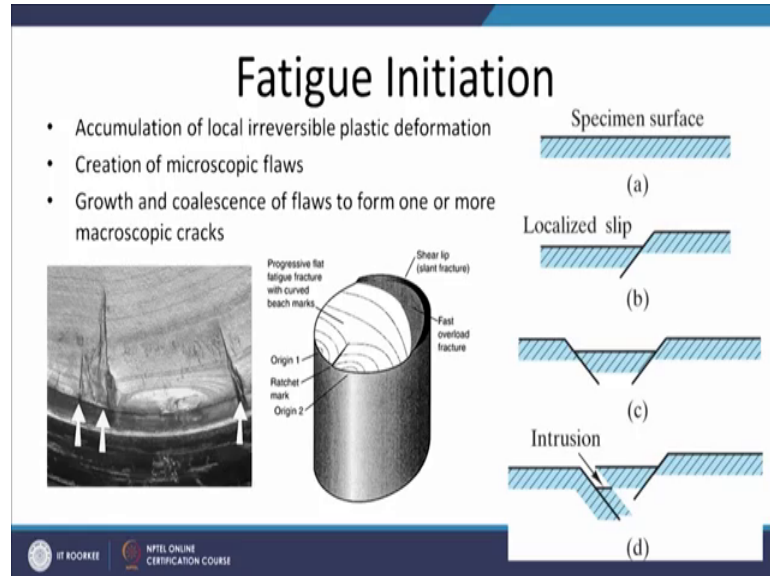
So, this is known as  $k_{th}$  or you can say the  $\Delta k_{th}$  the minimum threshold stress intensity factor range, which is required for crack to propagate and thereafter we have a diagram like this means the variation like this where the crack growth rate increases with the increase of the stress intensity factor range at particular rate at a fixed rate at a.

So, the rate will keep on increasing. So, this relationship is linear in its log scale and thereafter it is starts increasing. So, this is the commencement of the third stage, this is the second stage and this is the zone one where in the crack behaves as a non-propagating type. So, this is the second stage corresponding to which we write this equation  $\Delta k$  raise to the power  $m$ .

So, this is known as power law equation for the fatigue life estimation and we know the if we know that initially the crack size is of a given value, and we know the crack growth rate, then we can estimate the life of the component which has failed. So, now, these things will be shown with the help of a certain micrographs, now this is the diagram

which is showing that when the even when the component is very smooth under the localized slip conditions, there will be creation of the ups and downs at the surface.

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So, that will be leading to the creation of the intrusive and extrusion and these intrusions and extractions will be acting as a stress raiser and thereafter once the sub crack a stress raiser or the crack like defect has been formed of sufficient size, then the growth of the crack will be occurring in a particular way.

So, the zone where such kind of the crack initiation takes place that will have all two other different morphology as compared to the other cases, where other zones where the growth of where crack grows at a steady rate or the sudden fracture occurs.

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### Fatigue Mechanism

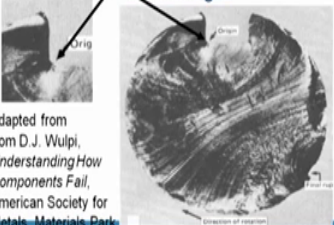
- Cracks in Material grows *incrementally*

$$\frac{da}{dN} = (\Delta K)^m \sim (\Delta \sigma) \sqrt{a}$$

typ. 1 to 6

increase in crack length per loading cycle

- To avoid failure  
 $K_{max} < K_C$
- Crack growth rate increases with
  - $\Delta \sigma$  increases
  - crack gets longer
  - loading freq. increases.



crack origin

crack growth

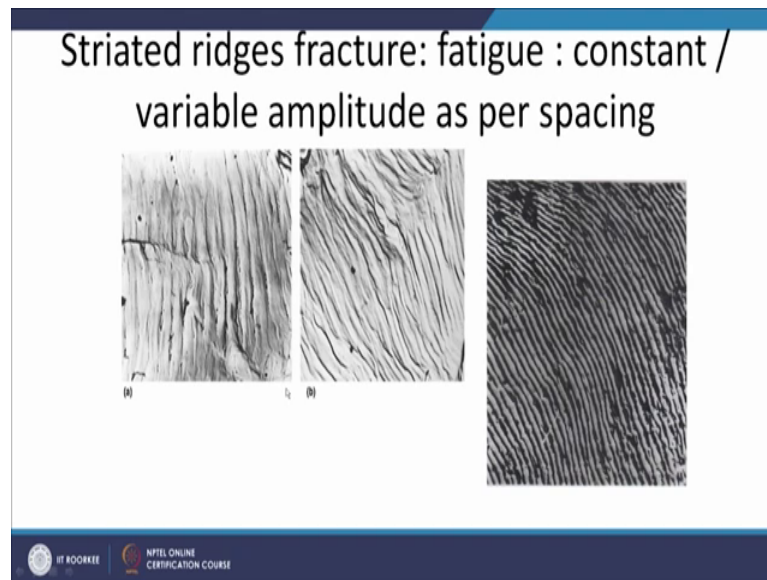
Adapted from from D.J. Wulpi, *Understanding How Components Fail*, American Society for Metals, Materials Park, OH, 1985

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So, this is what I have just explained that the crack growth rate, can be expressed using such kind of the equation which is known as a power law, and crack growth rate of course, increases with the increase of this stress range, which is clear from this diagram because delta stress range in as the stress range increases the stress intensity factor range also increases, which in turn increases the crack growth rate.

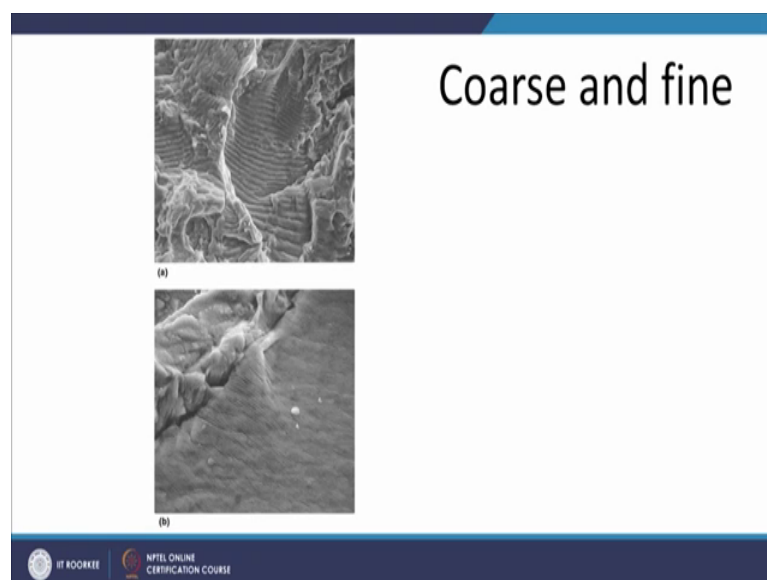
Similarly, as the crack size increases that then also the delta a by delta N will be increasing because here delta K is the function of the square root of a; a is the crack size for the open cracks and the half crack length in case of the internal cracks and increase of the loading frequency also increases the crack growth rate.

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So, as I was talking about that the striations are formed and which spacing between, which indicates that roughly the value by which the crack will be growing in each load cycle and the it can be constant or variable the constant stress amplitude results in the constant spacing otherwise it can be fluctuating for given uniform for the given material properties.

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And if there is a heterogeneity in metal properties of course, the spacing can also vary according to the kind of material through which crack is passing; the course these



striations can be coarse or fine striations will indicate that the crack is growing at a lower rate, while the courses is striations means the coarser or wider spacing between the striations, indicate that the crack is growing at a faster rate

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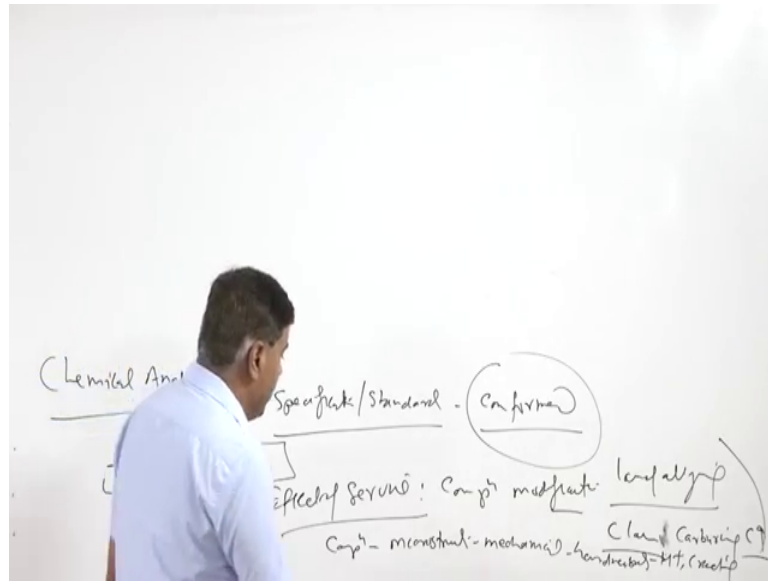
So, it is also possible, that it may we may not be able to identify where striations are present on the fracture surface it requires lot of efforts using the scanning electron microscope to establish the presence of the such a kind of the striations sometimes it also becomes difficult to locate where such a kind of the striations are present.

So, these are the examples of the poorly defined or no striation case even under the fatigue condition. So, the such kind of the lines will indicate the possibility of the striations, but these are not very properly defined on the fracture surfaces.

So, sometimes if the material is very complex in terms of the phases compositions, then and then it may not be possible to really establish the presence of this striations so clearly.

Now, another aspect of the failure analysis is about. So, is the chemical analysis. So, in the failure analysis procedure we have also to see the chemical composition of the component which has failed. So, there are certain things which we want to investigate with regard to the chemical analysis of the failed, component chemical analysis.

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Say the background information collected about the component if it says that the component is made of this particular metal. So, if we have the specification or the standard corresponding to which the component should be made so, that specification is to be conformed through the chemical analysis.

This is one thing that we would like to do like if this is the failed component, and if it was made of particular materials. So, we need to verify if really it was made of actually of that material or there was some variability. So, confirmation of the chemical composition is one thing that we want to do through chemical analysis, and minor variation although is not attributed to the deficiency in chemical aspects, but if the if the variation is really significant, then this can be one of the aspects because it affects lot of things related with the material structure, properties, cracking sensitivity etcetera.

So, the confirmation of the chemical composition is one thing that is what we want to see. The second thing if the component has been subjected to the effect of service effect of a service especially with regard to the compositional modification effect of service with regard to the compositional modification.

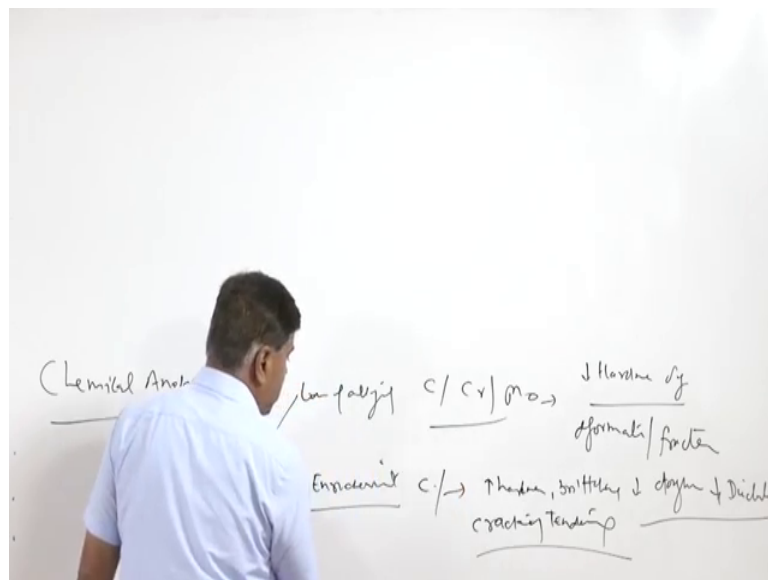
This can be there in form of the loss of alloying elements this is one thing, which we commonly see in terms of the like decarburization where loss of carbon takes place carbon loss takes place or carburization where the carbon content is increased.

Likewise high temperature exposure means exposure at a high temperature for prolonged period can also lead to the loss of the other alloying elements also from the component. And we know that the since the composition affects the micro structural aspects of the component which in turn affects the mechanical properties, also affects the harden ability and response to the heat treatment, cracking tendency all these are affected by the chemical composition.

So, it becomes important to really see if the composition was as per the requirement or it has been different from what has been what was specified or what was expected to be there in the component.

Or sometimes loss of alloying element or enrichment of the alloying elements in the component, during the service can also lead to the modification in properties which we can see with regard to the two aspects, one is like if the it is the loss of alloying elements then for example, loss of the carbon or chromium or molybdenum kind of elements from the surface, it will be leading to the reduction in the hardness reduction in the  $\sigma_y$ .

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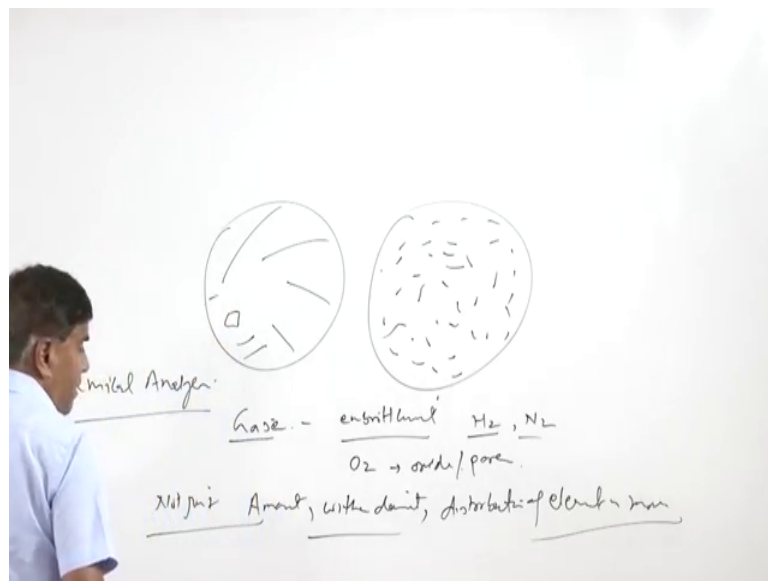
So, a reduction in the  $\sigma_y$  and hardness this can lead to the loss of strength and a loss of strength may lead to the deformation or even the fracture.

So, we need to really establish if there has been any losses or enrichment has taken place enrichment of the alloying elements and if addition of the carbon is taking place, then

like say during the carburization then it will be leading to the increase in hardness, increase in the brittleness reduction in toughness reduction in ductility. So, it is possible that that under the impact load condition such kind of the components can fail in very brittle manner. So, this will be increasing the cracking sensitivity or sensitivity for the fracture in presence of the crack.

So, it is important to consider this aspect especially with regard to the macroscopic study of the fracture surface whether it has been ductile or brittle accordingly it can suggest the possibility for the loss of alloying elements. Not just this it is also important to it is not just important to find out the numerical value of the chemical elements present in a given component, but also we need to see what kind of the gas is present because the gases can lead to the change in the properties in form of embrittlement.

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Embrittlement of the like presence of the hydrogen, nitrogen in iron leads to the especially high strength steels it leads to the embrittlement.

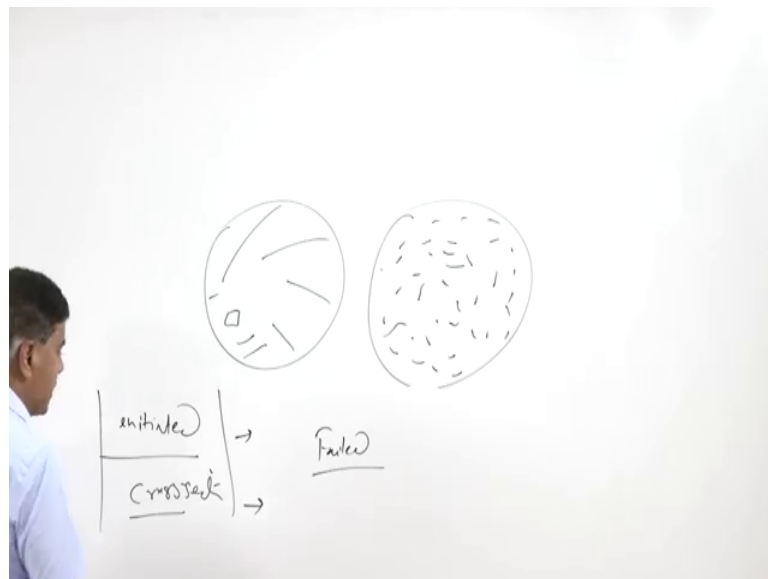
Similarly, the presence of the oxygen also leads to the oxides or the pores in the material. Apart from determining the not just amount, not just amount is amount of the alloying elements or the elements present in the metal are important within limits of the variation in amount, the distribution of the alloying element is more important distribution of the elements is more important. Because like say if this is the metal and if most of the things

if a particular element has been segregated like this in particular location, then it will not be able to offer its effect in the entire mass of the component.

While if the same one has been well distributed in the matrix then it will be able to offer, the desired change in the microstructure desired properties in the components. So, homogeneity is more important than the exact amount, but of course, the amount variation is has to be within the limits.

So, it is important therefore, to see the kind of distribution of the alloying element which is there in the field component, and for that what will be the zone of interest of course, the where from the fracture has been Initiated or the cross section where failure has taken place.

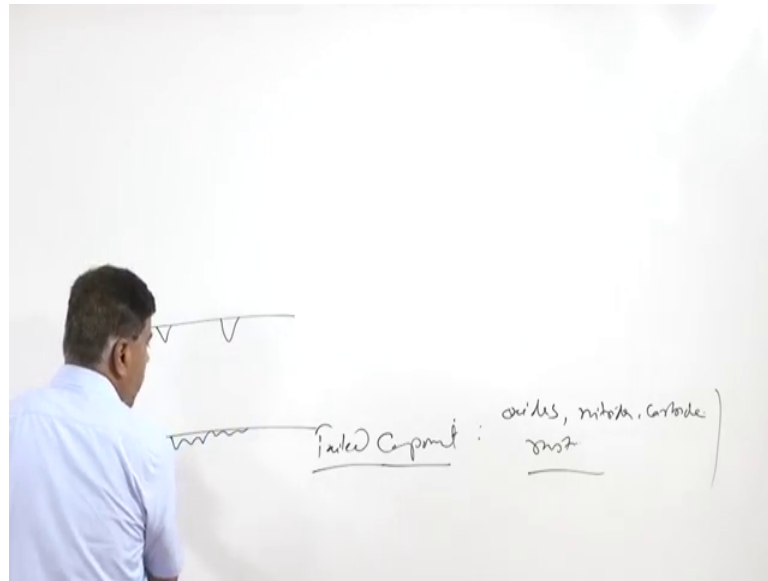
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So, of course, we need to take the samples from the fracture initiation site or the location near the fracture surface. So, that their chemical composition can be established to see, if there has been a segregation of certain kind of the elements gases or not.

So, this is one thing apart from this the segregation distribution related aspects of the alloying element, we need also to see if the failed component has been exposed means has been in the service for a longer period, then the service conditions will also have effect on the surface characteristics of the component, which may be they are in form of the formation of the oxide.

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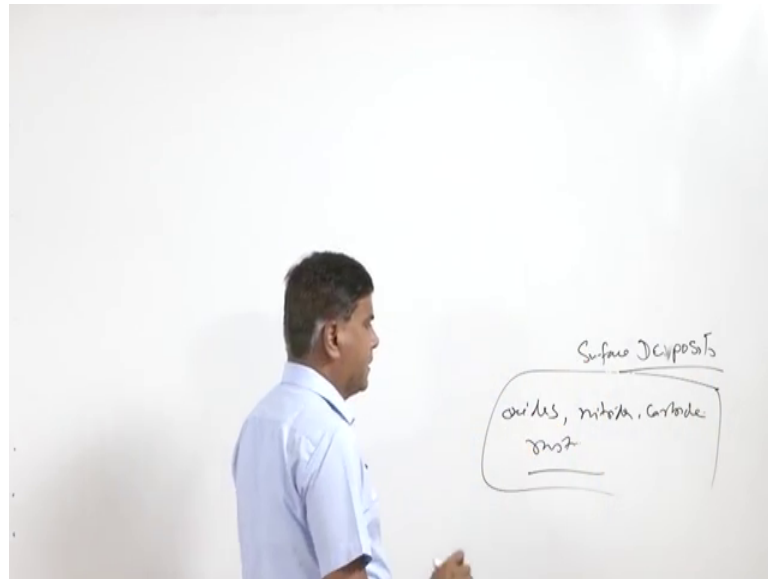


So, the like failed component after prolonged use can show the presence of surface deposits in form of the oxides, nitrides, carbides some shoot formation rusts etcetera.

So, all these things if these aspects have deteriorated the properties or they have created some stress raises on the surface of the component, they have if they have eaten out the material uniformly from the surface or they have caused the localized damage in the component this may be in form of pits or crevices or presence of some causing the development of the irregularities on the surface.

So, now these are things which are presented the surface as a due to the conditions which have been exposed or experienced by the component during the service, these need to be these surface deposits need to be investigated deposits need to be investigated.

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So, whatever is present at the surface and if it has affected the surface properties irregularities proper mechanical properties of the component at the surface, all these need to be investigated. So, the impurity is presented the surface also should be investigated.

Why it is important? Because the chemical analysis has the direct effect on the microstructure, which in turn affects the mechanical properties also affects the heat response to the heat treatment.

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## Chemical Analysis & FA

- It affects
  - Microstructure
  - Mechanical properties
  - Response to the heat treatment
  - Corrosion behaviour
  - Hardenability and cracking sensitivity

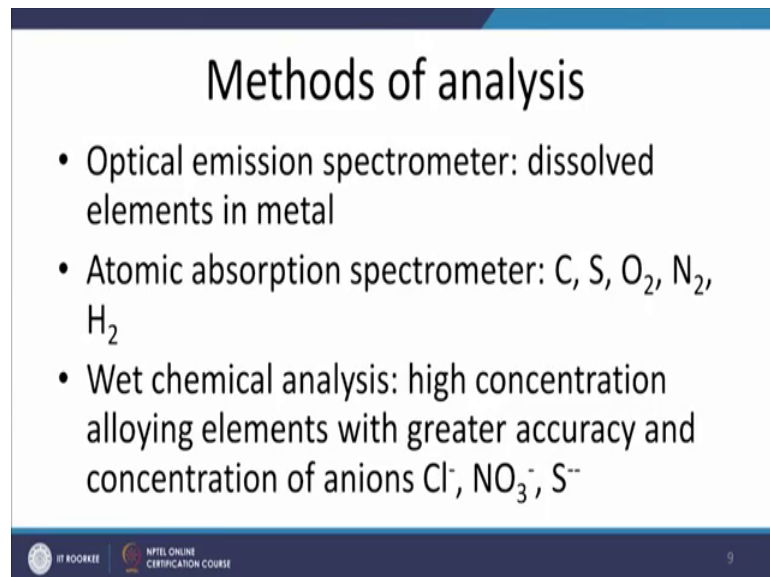
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And not just that the corrosion resistance of the material is also affected because if the homo composition is homogeneous, then corrosion resistance will be good if there is segregation of some of the alloying elements either the grain boundary or within the grain, then it will be forming the galvanic cells easily and which in turn will be reducing the corrosion resistance.

Similarly, the higher concentration of the alloying elements will be leading to the increased harden ability in case of steels, and which in turn will be increasing the crack sensitivity.

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**Methods of analysis**

- Optical emission spectrometer: dissolved elements in metal
- Atomic absorption spectrometer: C, S, O<sub>2</sub>, N<sub>2</sub>, H<sub>2</sub>
- Wet chemical analysis: high concentration alloying elements with greater accuracy and concentration of anions Cl<sup>-</sup>, NO<sub>3</sub><sup>-</sup>, S<sup>-</sup>

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Now, there are various methods, which are used for the chemical analysis purpose depending upon the purpose what is to be analyzed in what size what quantity which material is there to be analyzed, we have the different things like optical emission spectrometry is used for determining the elements dissolved in the metal.

Then atomic absorption spectrometer is used for determining the elements dissolved in the metal as well as and most commonly it is used for determining the carbon sulphur content apart from the other alloying elements also it is also used for determining the concentration of the oxygen nitrogen, hydrogen.

Then wet chemical analysis is used for determining the concentration of the alloying elements which are higher in concentration. Elements which are high in concentration



can be established accurately with the bread chemical analysis, the same time it can also be used for determining the presence of anions like chlorine chlorides nitrites and sulfides.

Then other methods of the spectroscopy like the x ray diffraction based spectroscopy is primarily used for the crystalline materials, and x ray fluorescence spectroscopy is used for both crystalline as well as the in amorphous materials, which can be applied for both solid liquid and the gases. Then infrared ultraviolet infrared or ultraviolet spectroscopy can be used for determining the composition of the organic compounds such as oil grease rubber and plastics.

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**Methods of spectroscopy**

- X ray diffraction based methods for crystalline compounds
- X ray fluorescence spectroscopy for both crystalline and amorphous metals: solid, liquid and gases
- Infrared/UV spectroscopy: organic compound such as oil, grease, rubber and plastics

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## Micro Probe Analyzer

- EPMA: surface area of about 1 microns in diameter up to 0.1 % content up to atomic No. 3 elements
- Ion Micro Probe Analyzer: same as EPMA with element as low as 100 ppm

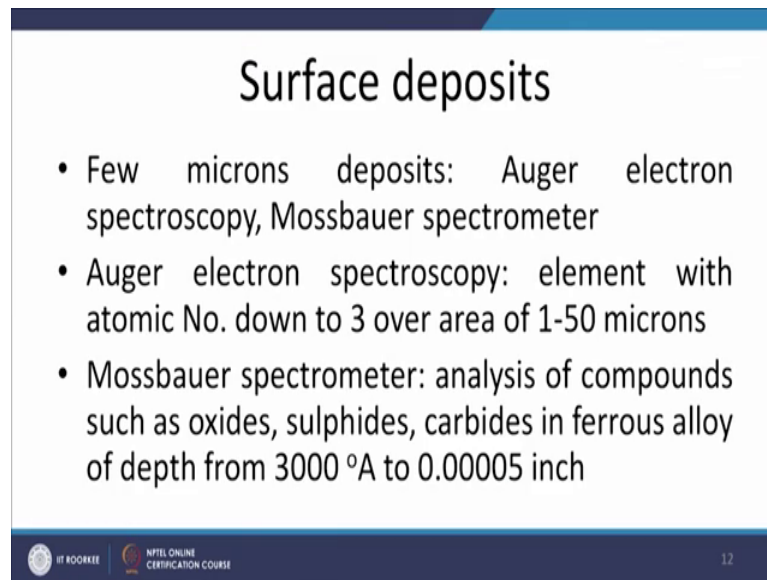
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Then there are two good approaches which are used for determining the distribution of the elements in the metal like EPMA which is called electron probe micro analyzer or ion micro analyzer this is. So, EPMA is used for elemental analysis over of the surface of the diameter of one micron for analyzing the components analyzing the elements down up to the three atomic number and the content is up to 0.1 percent.

So, the me the this is the threshold or resolution of such device where the elements greater than 0.1 percent of the atomic number greater than 3 can be analyzed using this approach and it analyzes the area of about 1 micrometer.

Then ion microprobe analyzer is same as that of the EPMA, but it has much lower resolution whether regard to much better resolution as compared to that of the EPMA because it can analyze the elements, which are present as low as 100 ppm.

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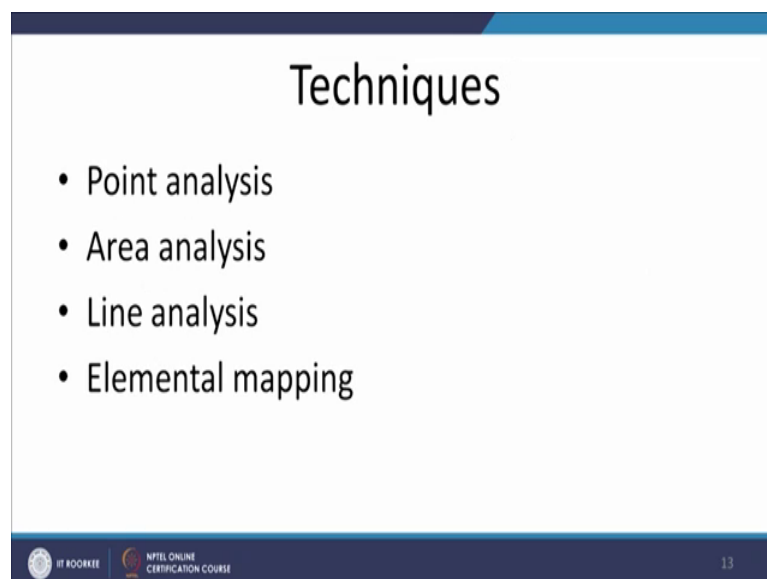
## Surface deposits

- Few microns deposits: Auger electron spectroscopy, Mossbauer spectrometer
- Auger electron spectroscopy: element with atomic No. down to 3 over area of 1-50 microns
- Mossbauer spectrometer: analysis of compounds such as oxides, sulphides, carbides in ferrous alloy of depth from 3000 Å to 0.00005 inch

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Then for analyzing the surface deposits there are two techniques. So, Auger electron microscope is used for determining the for measuring the elements up to the atomic number 3 over the area of 1 to 50 micrometer, while the mossbauer spectrometer is used for analysis of the compounds are like sulfides carbides and oxides present in the ferrous systems and which can analyze the depth up to three from depth of varying from 3000 angstrom to the 0.00005 inch.

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## Techniques

- Point analysis
- Area analysis
- Line analysis
- Elemental mapping

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And then there are two techniques which are used for there are a few techniques which are used for the analysis purpose and for most of these techniques, work in combination with the.

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Scanning electron microscope for like EPMA or FESEM or ion electron mic ion microprobe analyzer these work in combination with the sam. So, we need to magnify the sample to be analyzed sufficiently so, that we are able to identify and recognize the phases which are to be analyzed.

So, like say A B C D these are the four phases present in the sample, and if we have to analyze them we can follow certain techniques and these are called a point analysis. In case of the point we just select a particular point of way for the analysis and it will give us the and the percentage of different elements present in that at that particular location or, but that particular point. So, here what the this phase is made of that will be indicated by analyzing the very small area like a point.

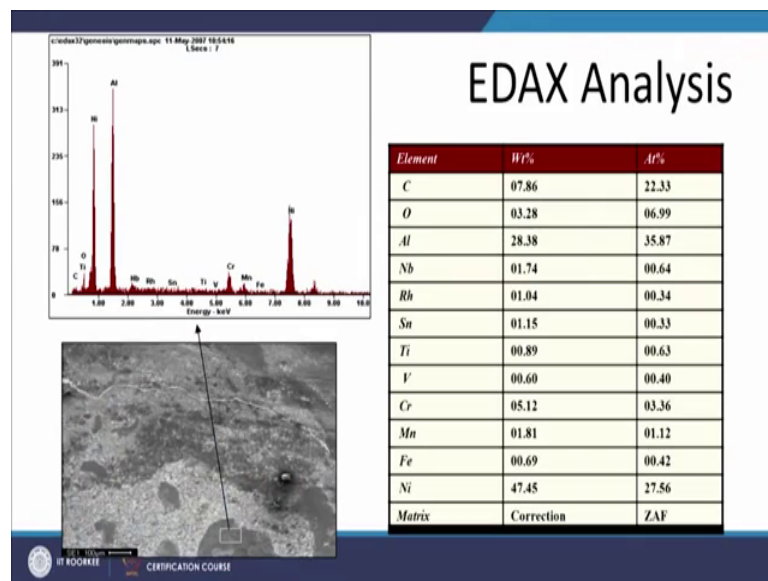
But if we want that know we need to have the better representative zone of the area being analyzed. So, we can select a particular area like this and then it will be analyzed for area analysis and know if you want that know we have to have the variation in the composition varying from one zone to another. So, then we can draw one line between the two points; the points of our choice and then we can draw one line like this and if

there is a falling if there is something present at the grain boundary that it also can be analyzed, what is done in this case?

We select the distance to be analyzed between the two points and then we also select the interval of the interval at which we the different points need to be analyzed. So, this will be giving us the idea about the different the variation in the concentration of the different elements, on moving from location one to the location another this one is called line analysis.

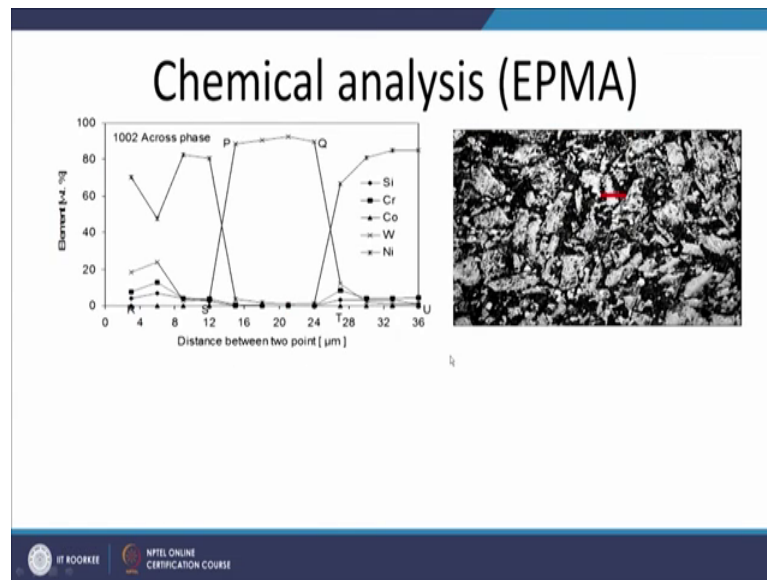
We may also be interested in that where what is present and how it is distributed over given area. So, for that purpose we also use the elemental maps. Elemental maps will be able to highlight where what is present which element where it is present and what is the kind of the distribution, within the grain or away from the or near the grain boundary. So, this is what has been shown in this in these two three diagrams here this is the.

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This is the EDAX analysis which is carried out with the help of feseem and here this area is analyzed to see what is present in what quantity. So, this is the kind of analysis of the different elements of this particular area.

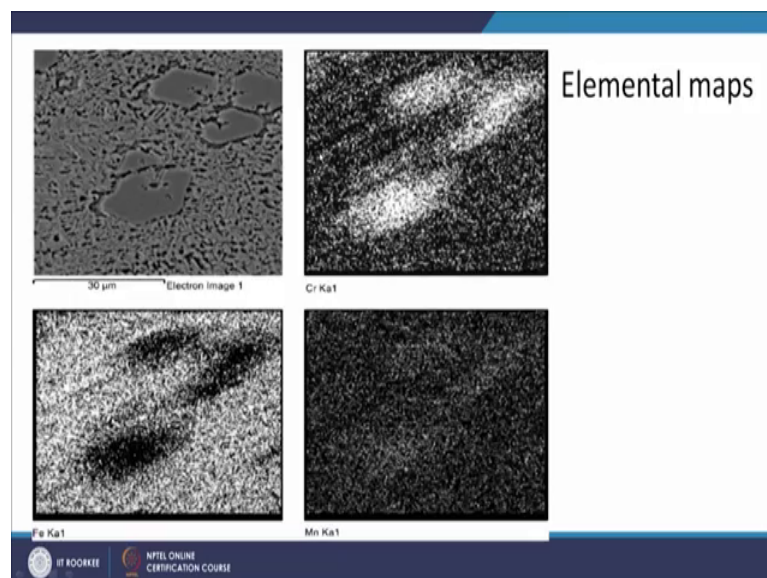
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Now, we will see this is the line analysis where that the between two points one line is drawn and the analysis is carried out between these two points and the distance between the points which are to be analyzed can be selected suitably.

So, what it shows that this white particle having the higher concentration of the tungsten this is what can be seen here while at this location it has a lower concentration of the other elements like the nickel. So, nickel is high in the matrix and the particle is made of the tungsten.

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So, such kind of the things can be done for the line analysis and then it is the elemental mapping.

So, we need to choose the location which is to be analyzed. So, this is the matrix these are the particles which are being analyzed and these particles are being analyzed for represents of the different elements like this one shows the chromium con and distribution of the chromium. So, what it is showing that, wherever at these particles wherever the bright particles are present indicating the presence of chromium and wherever dark dark zone exist indicating the presence means absence of the chromium or a presence of less chromium.

Similarly, this one shows the distribution of the iron what it is showing that matrix is a dominated with the iron, and in these particles the iron is in very limited quantity. So, wherever iron is missing or it is less in concentration that zone will be shown by the dark area. Similarly the for the manganese content almost to the distribution of the manganese is somewhat more means the manganese content is somewhat more in these particles as compared to that in matrix.

So, wherever if the particular particle is present in greater quantity that location will look brighter I will say it will look the darker one. So, this is how the elemental list a maps can be used to see where what is present um.

So, now I will summarize this presentation, in this presentation basically I have talked about the how to determine the fatigue fracture in using the suitable techniques like microscopy and microscopy and also I have talked about the importance of the chemical analysis in failure analysis and what are the different techniques which can be used for carrying out various types of the chemical analysis

Thank you for your attention.