Fundamentals of Surface Engineering: Mechanisms, Processes and Characterizations

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Lecture-59
Characterization of Modified Surfaces: Metallography

Hello, I welcome you all in this profession related with the subject fundamentals of surface engineering and you know now we are talking about the characterization of the modified surfaces which have been engineered through various techniques for improving the mechanical properties and tribological resistance so that they can offer the longer tribological life of the component during the service.

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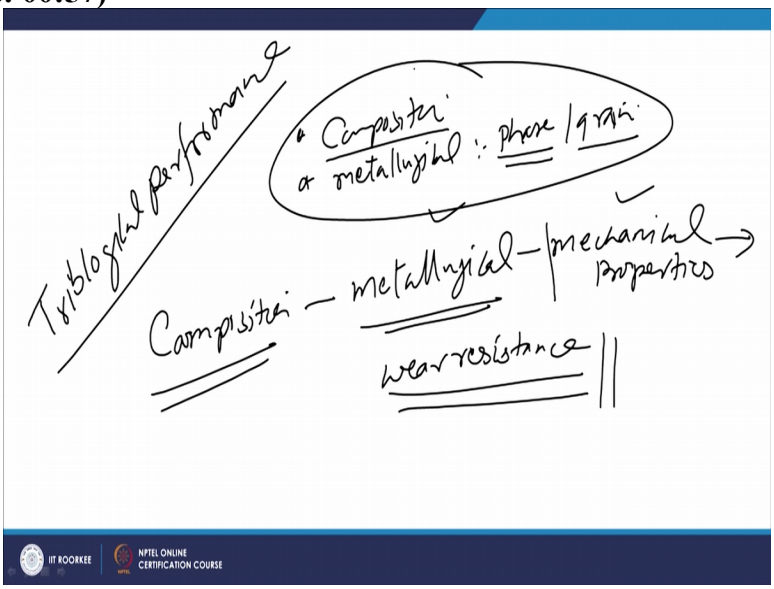

But this tribological performance of the component is influenced by the various characteristics which includes like the composition of the given material will be determining the kind of the metallurgical properties it will be having in terms of the phases, the grain size, the structure etcetera and the metallurgical properties affect the mechanical properties of the surfaces which have been engineered and the variation in these mechanical properties eventually will be leading to the change in the wear resistance.

So, this is the kind of the link which exists if you want to change or the regulator improve the wire resistance for a particular kind of the service conditions then we need to adjust the properties accordingly and which can be done by regulating its metallurgical properties and the

composition in changing the metallurgical properties of place a big role. And therefore it becomes important to study both the composition of the modified surfaces. If the chemical composition modification methods have been applied or the some fusion based surface modification technique has been used.

Where there is a possibility of change in composition or the coating are the weld surfacing or the cladding kind of things have been applied when we need to see really what is exactly has been applied on the surface of the substrate so that we can a particular kind of metallurgical properties and according to the mechanical properties for improving the wear resistance. At the same time for a given composition as per the thermal and mechanical history of the modified surfaces.

We may have the different kind of the metallurgical properties which we primarily see in terms of the phases it has and the kind of the grain structure it has. So basically we see under the metallurgical properties phase structure and grain structure of the material. So, this lecture primary focusing on these two aspects how to study the composition of the modified surfaces and how to investigate or study the metallurgical properties of the modified surfaces in this regard if we see rationale behind studying the chemical composition and the metallurgical properties of the material.

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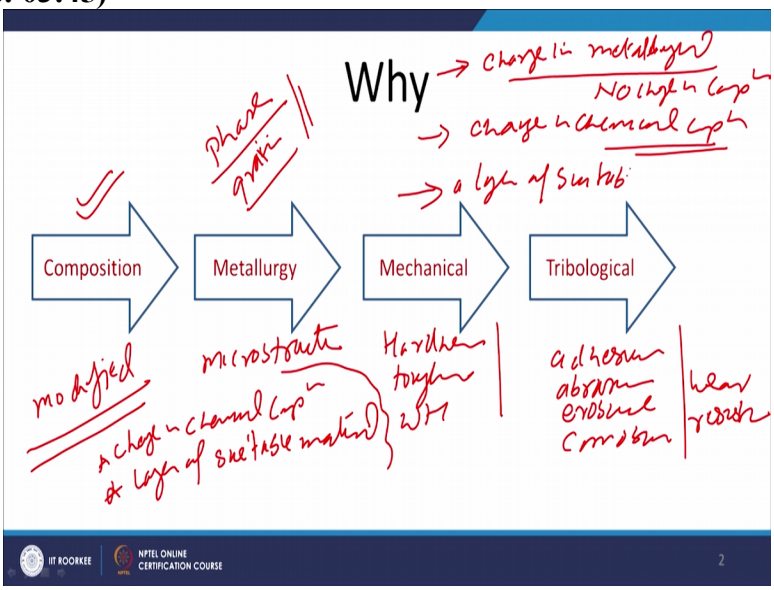

So, these slide shows that for a given modified surface of a particular composition will have a set of the metallurgical properties with regard to the microstructure especially and this microstructure will be governing the mechanical properties such as hardness, toughness, work

hardening tendency. And these factors intern will be governing its adhesive, abrasive, erosive and corrosive wear resistance corrosive wear resistance.

So, in order to link these behaviours we need to study the chemical composition if the chemical composition modification methods have been applied in where the change in chemical composition of the surface methods has been applied. Or if we have applied a layer of the suitable material layer of suitable material has been applied on to the surface of the substrate. So, up to what extend the chemical composition of the modified surfaces have been realised.

That is what we try to see under this is stage of the studying the chemical composition. And once if it has been modified and what kind of the phases have been obtained and what is the grain structure of that layer which has been modified through suitable surface modification techniques. We know that there are three broad categories of the surface modification techniques one is where just the change in metallurgical properties is brought in without changing the chemical composition.

Just change in the metallurgical properties no change in composition is brought in and the second one second category of the approaches of the surface modification is where the change in chemical composition is used to certainly the study of the chemical composition becomes strong case for this group of the methods. Similarly in third category where are there of suitable material is deposited on to the surface of the substrate so that the required improvement and properties can be achieved.

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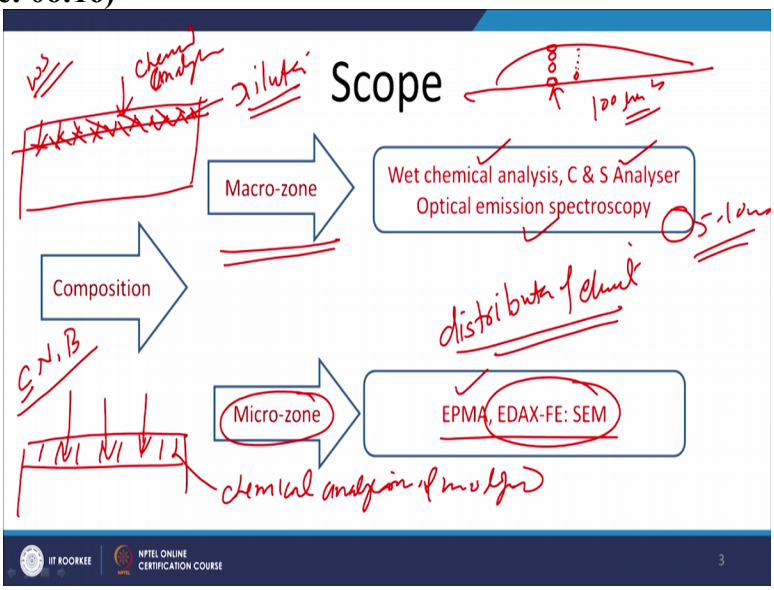

If you have to study the chemical composition of the modified surface say this is the substrate and its one layer has been modified through the suitable technique like just changing the surface metallurgy or changing the chemical composition or building up of a layer of a suitable material. So, as per the process; like if you are using carburizing or nitriding, boronising in all these cases carbon, nitrogen or boron is introduced. So, what is the extent of the depth what is the depth up to which elements have been introduced.

And what is their weight percentage is which have been introduced to see the effectiveness of these methods. So, for this purpose we need to carry out the chemical analysis of the modified surfaces. Similarly if we have build up a layer of the suitable material like over the surface of the mild steel we have deposited layer of the austenitic stainless steel. So, due to the melting of the base metal there may be changed in chemical composition of the austenitic stainless steel which is termed as dilution.

So, in order to see the extent of the dilution which has taken place due to the fusion of the base metal in the weld surfacing processes again we need to perform the chemical analysis of the surface. So, for the two categories of the processes where a layer of the metal is deposited or the chemical composition is intentionally modified to enhance the properties.

We need to definitely applied to the chemical we need to do the chemical analysis so that we can assess the suitability of the modified surfaces for a given application as far as the extent of improvement in the properties in terms of the mechanical properties or the tribological properties which have taken place.

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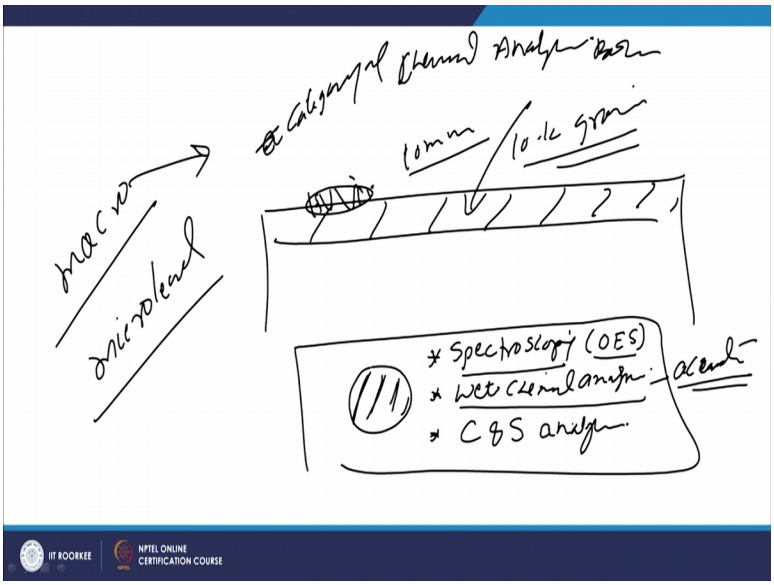

There are two broad aspects related to the chemical analysis one is like macro level analysis and another is macro level analysis. So, in case when a surface which has been modified like this so for macro level analysis we select one big area like say maybe of the 10 mm in diameter which will be having the 10's of the 1000's of the grains are the micro constituents. So, average composition of that particular zone is achieved and it indicates the average composition of the modified zone.

So, for this purpose one category we use one category of chemical analysis processes. So, when very large area of the modified surface is covered like this is the area which is covered for the chemical analysis so that it gives the better representative composition of the elements present in modified surface. For this purpose we can use the different methods one is the most commonly used is spectroscopy.

Most commonly it is written as OES Optical Emission Spectroscopy then wet chemical analysis method for wet chemical analysis from this entire area we have to deal out the chips and the large volume of the material in form of the chips is used for chemical analysis. By dissolving the material to be analysed in suitable chemicals this kind of analysis is performed perform and this gives very accurate results as per as the analysis is concerned.

And then there is another kind of the spectrometer which helps in determining the carbon and sulphur concentration in especially in steel so that is why it is called carbon sulphur analyser. So, this is one type of the analysis where 1000 of grains of the constituents present on the surface of

the modified component are used for the analysis. And it covers the macro zone very large area of the modified surface is used for the analysis of the chemical composition.

In case of the micro level analysis is done of the micro constituents individually or over a particular zone for micro level analysis and the kind of techniques which are used for the micro constituent's chemical analysis of this micro constituents those techniques are different so that is what you will see here for macro zone analysis when the large area is covered can use the wet chemical analysis carbon sulphur analyser and optical emission spectroscopy.

And very large area of the sample to be analysed is used maybe it can vary from 5 to 10 mm including 1000's of the micro constituents present in the surface of the modified component. When we have to when it is required to perform the chemical analysis of the micro constituents individually or over a very small area that is called like the chemical analysis of the micro zones and for that purpose we can use the electron probe micro analyser or energy dispersive x-ray analysis with the help of FTSEM.

So, when are these two techniques are used these two techniques are extensively used for the research purpose and to see the effectiveness of the surface modification the micro zone analysis using EPMA or EDS helps a lot to see a presence or distribution of elements present at the different zones at micro level. Say for an example this is the modified zone and we want to see that what are the elements present at near the interface than on approaching towards the surface.

How the composition of variation is taking place then in that case we need to select this 4 different areas and analysis of each area is to be performed and this area may be very small like 100 square micrometre micro metre square. So, very small area is analysed for these purpose we can select even small point also does not have large area and this point may be of diameter of 1 micro meter, 2 micro meters. So, there are various techniques for performing the micro zone analysis using EPMA or EDAX.

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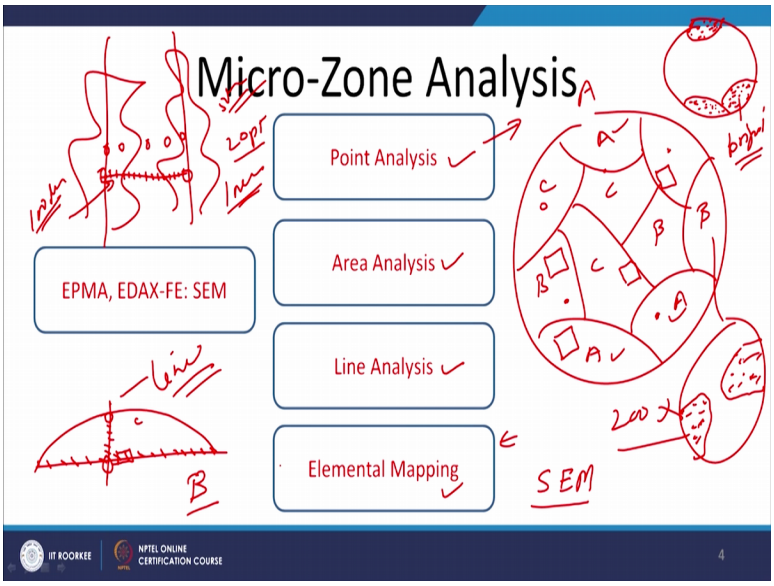

And these techniques are these are the four techniques which are used for micro zone analysis 1 is called point analysis and another is area analysis 3rd is line analysis and 4th is element analysis and each one now we can I will try to understand like say this is the area which is under focus at a magnification of say 200 X. So, we are able to see various micro constituents under the scanning electron microscope.

So, these EPMA and EDAX are both are used in combination with the scanning electron microscope and we select the suitable magnification so that the micro constituents are very clear. If these are various constituents which are present in the micro structures likes A, B, C, D, A like this we can go for marking a different micro constituents are present. So, if you want to see how the compositional variations is taking place within the grain and going from one to another.

Then we can use this kind of various approaches of the micro analysis like say if the analysis of particular small zone is to be carried out then we select particular point and it is analysed. This may be of just like small area of 1 micrometre 2 micrometre in diameter. In point analysis we select the point which is to be analysed it may be grain boundary or it may be a particular micro constituents which is of smaller size.

So, for point analysis we select the point on that particular micro constituent which is to be analysed and that will be giving us the result about the kind of elements which are present at that particular point. For area analysis we select the larger area as compare to the point for the purpose of analysis so in that case one area like this is selected and it is analyse this area maybe at the centre of the at the grain boundary or within the grain or close to the grain boundary.

So, that area is chosen as per our interest which area is to be analysed. For example in the surface modification when we have developed one flame spread coating and this is the substrate are the base metals and is the coating which has been developed. And we expect that there is segregation or localisation of some of the elements near the interface. Now we can we can select this area and it can be analysed.

If you want to see how the concentration of particular element is wearing from the interface to the surface of coating then we select these two points and then scanning is performed all along this line to see how the compositional variation along that particular line taking place. So, for that purpose we have to carry out the line analysis. So, line analysis helps us to see how the concentration of the how the distribution of the element is changing between the two selected points of our interest.

Say this is one grain and this is another grain and if you are interested the kind the way by which the distribution of the element is changing from the ones centre of the one great to another then we can choose this 2 points are connected through line and say this distance is of the 100 micrometre. Then time interval between the any two between these two points means the spacing of for the other points between these two points along a particular line can be selected.

For if you want to perform the analysis at 20 points between these 2 points which are is located at 100 micrometre distance then the 20 different points will be analysed by the EDX or EPMA at the interval of the 5 micrometre. So, and if you want just to see that analysis of the selected points is carried out then we can simply carry of the point analysis. And if you want to see the line and scan or if want to see the continuous variation in the concentration of the elements between any two points than we have to perform the line analysis.

The last one is the elemental mapping; elemental mapping helps us to see where which element is present in selected microstructure. Say if a according to this schematic diagram of the microstructure ABA ABA the same types of the micro constituent assembly here AECC are made of the different micro constituent the different phases they will be made of the different micro constituents.

So, if the mapping is carried out elemental mapping is carried out for this entire area say mapping for the A is carried out first then A will be giving us all these grains where A is present

these areas will be bright like if the mapping for A is being done then wherever A is present it will be showing the higher concentration of the A and these areas will be bright. Rest of the areas where A is absent that will be shown as dark field.

Similarly when the B is analysed analysis for the B element sown as dark field similarly when the B is analysed analysis for the B element is done then what will see that the certain zones we will B is present they will be bright while other locations will be dark. So, this is the location where B is present so these fields will be bright. The elemental mapping gives the areas zones where particular element is present. So, it helps to map the presence of particular element in given micro structure.

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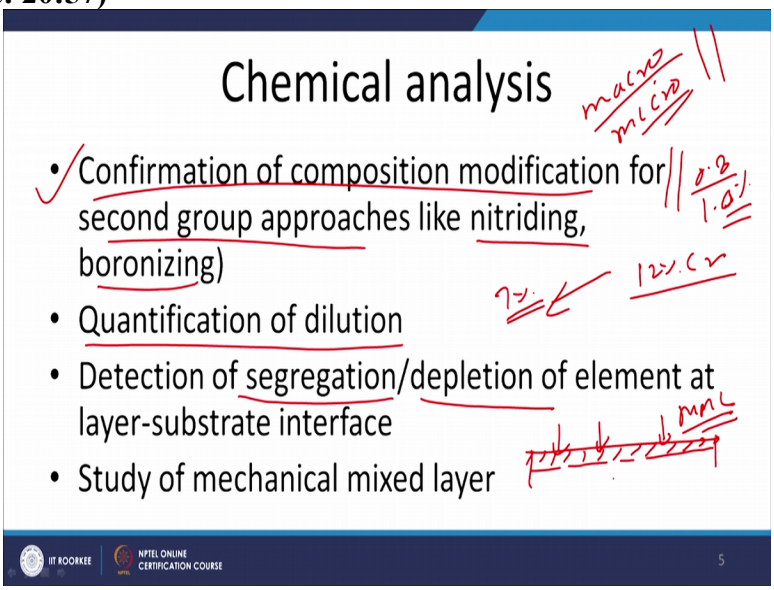

So, when whether we are carrying out the microphone analysis are the micro zone analysis in both these kind of analysis help us in understanding or in confirming the composition of the modification, confirmation of the modification. Say if you have carried out the carburizing. Then carburizing is carried out to increase the carbon content from the .2 to.8 or 1% of the carbon content and weather and this we can confirms through the chemical analysis if the carbon content has been increased to the .8 or 1% level or not.

So, out after the surface modification if the required chemical composition has been achieved or not that is what we can from through the chemical analysis so, confirmation of the chemical composition modification for the second group of the approaches where we use carburizing nitriding, nitriding or boronising. So, we can analyse to see whether the boron is present in the

required amount or not or the nitrogen is present in required amount of not. So, that it will be forming the different phase's compounds for improving the properties.

Then the quantification of the dilution it also helps to analyse if we have Adele like 12% of the chromium but actually in the weld surfacing it is just 7% so, reduction is attributed to the dilution. Similarly we can see whether any kind of the segregation are the laws of the alloying elements are the depletion of the following element is taking place especially and near the coating substrate interface or any other zone of the modified surfaces.

So, it helps us to see the distribution of the elements in the modified zones and the components which are subject do adhesive wear. We know that a typical mechanical mixed layer near the surface is formed which will be having the elements from the counter surface as well. So, what is the depth of which kind of the modification chemical composition modification has taken place and what are the different elements present in the modified zones that is the mechanical mixed layer?

What are the different elements present in mechanical mixed layer that is what can be analyzed to the micro analysis of the worn out components. These are the 3 or the 4 purposes of the chemical analysis with regard to the surface engineering.

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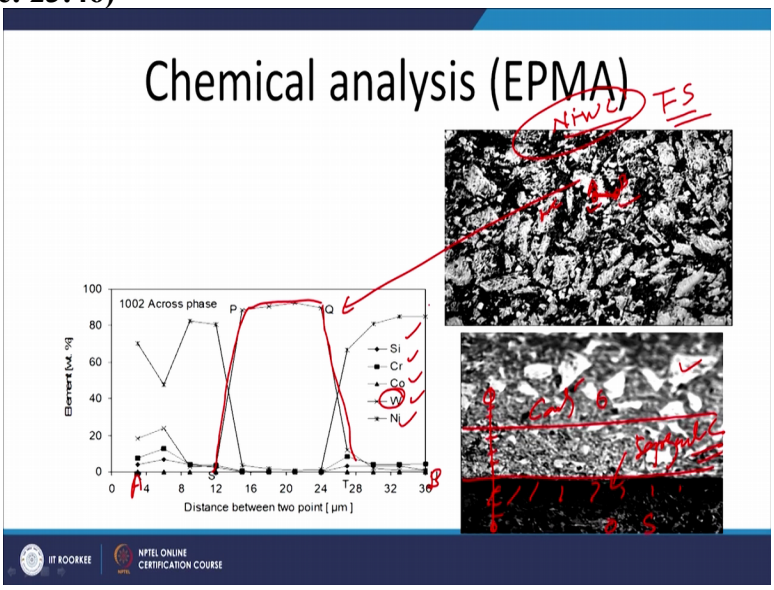

This is an just an example of the line scan analysis like say this is the Nickel tungsten carbide coating deposited by the flame spraying method and the scooting is having primary tungsten carbide and other dark one is the eutectic matrix. So, here the analysis is performed between

these two points like say the points A and point B. So, considering this point A is here and point B is here.

And what we see that when the scanning is performed as the line scanning is done along with white bright particle there is a huge increase in the concentration of the tungsten which is suggesting that these white particles are enriched with tungsten coating tungsten carbide particles were used. So, EPMA make informed that these bright particles are polyhedral shape bright particles are of the tungsten carbide particle.

Similarly here we can also do the line scan in this case this is the substrate and this is the coating substrate bond interface and this is a coating. So, we can we can see here at the interface near the interface the concentration of these carbon particles is less. And if we see there is a dark band so this is this kind of the band is formed due to the segregation of the carbon near the coating substrate interface and this zone may be hard.

And once if the heat treatment is performed Sometimes this zone is eliminated by the diffusion of the carbon into the substrate. So, basically EPMA analysis helps us to see the distribution of the different elements along a particular line. Here also we can perform this kind of analysis starting from substrate to the coating we can perform the line scan along this particular line. It will be showing the variation in the elements that we want to analyse.

And this kind of analysis can be performed for the different elements as per our choice. For example in this case the analysis between these two points was carried out for Nickel tungsten Cobalt Chromium Silicon. So, analysis elements for analysis are chosen in such a way that they are present in that particular material on that particular coating.

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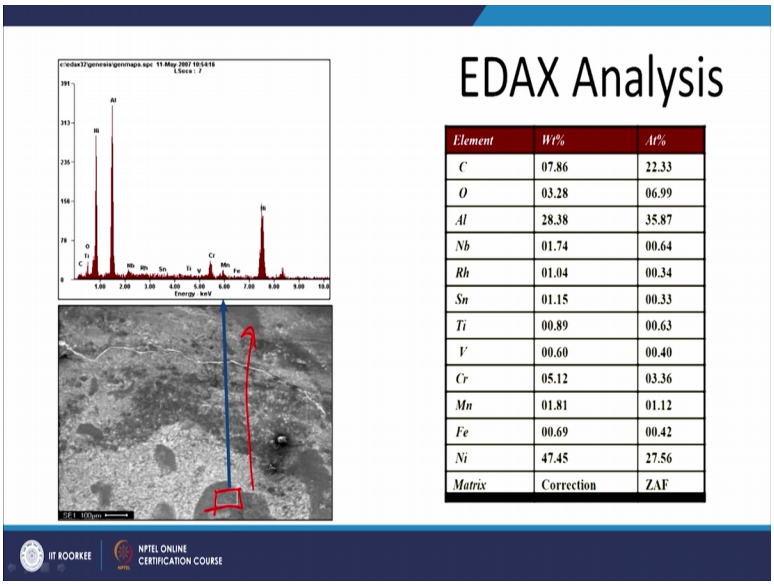

Similarly this is an indicator of the aerial analysis, for aerial this is this is the area which was selected in it was analysed this is showing that diffraction pattern achieved through the EDX analysis.

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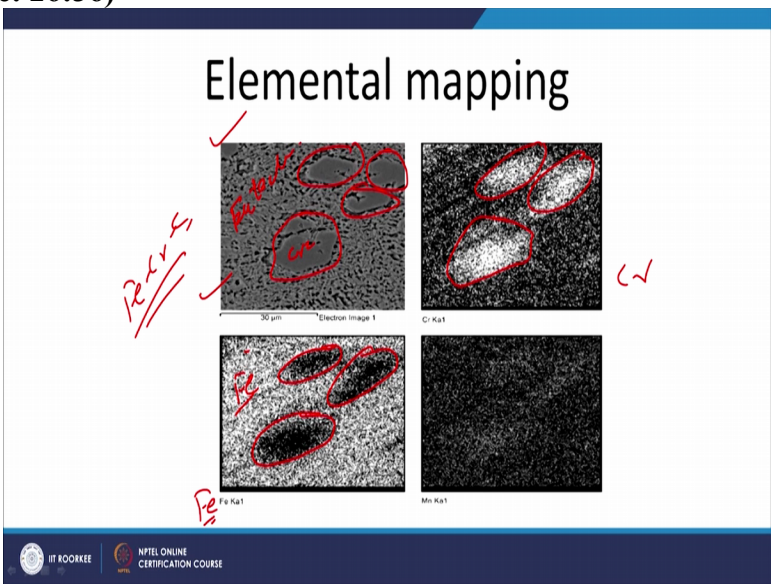

I was talking about the elemental mapping like say this is the microstructure of the hard facing coating where the matrix is of the eutectic and it is having the carbide particles like chromium carbide particles. Wherever chromium carbide particles are present like this elemental mapping is carried out for chromium will see the bright particles in the zone where is present. And other locations and when the analysis done for the other is this was the iron chromium carbide weld surfacing.

In this case one the matrix primarily is comprising the iron where the bright particles are present and these chromium carbide particles where chromium was dominating iron is deficient should we are getting the dark one. While the manganese is almost dark or we can say manganese is well distributed in the entire zone.

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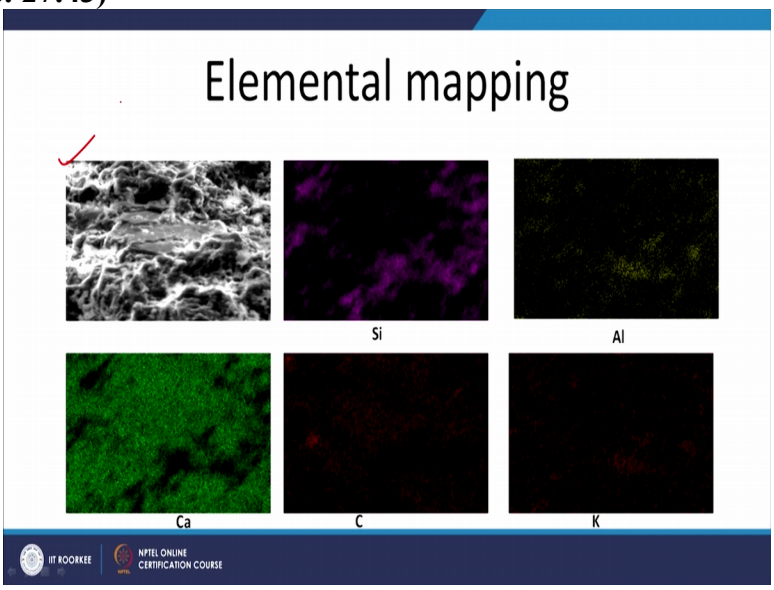

Similarly this is the elemental mapping for another and kind of the fracture surface which is showing where the silicon is present where Aluminium is present where carbon is present sodium or potassium is present and calcium is present. So, elemental mapping helps us to see for a given microstructure it helps to see where which element is present. So, elemental mapping helps us to see for a given microstructure it helps to see where which element is present.

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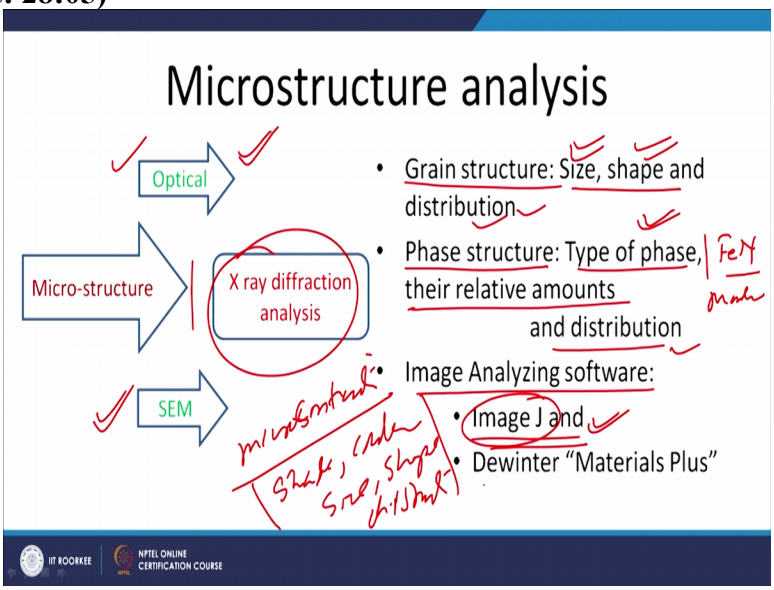

Now and other important aspect is the microstructure and microstructure is study we can carried out we can carry out using the optical microscope scanning electron microscope. So, for the low magnification or for less resolution purpose is making you can carry out even the optical microscope. Otherwise it is performed using the electron microscope scanning electron microscope.

But microstructure simply indicating the different micro constituents which are present they do not confirm which phase it is. It will be just showing the different shades colours different sizes and different shapes and their distribution these are the three things. It does not confirm which type of the phase particular thing is. For confirmation of phase we carry out the; we need to carry out the X-ray diffraction analysis.

So, there are two broader aspect as per as the microstructure analysis is concerned one is the grain structure study where in we have to see the grain size shape of the grains and the distribution of grains means where which size and shape of the grain is present from the interface to the surface of the modified coating. Similarly in the phase structure we try to see what are the different types of the phases are present in the modified zone.

And then their relative amount and how they are distributed. Like for the night riding there maybe iron nitrate formation in case of the carburizing it may be martensite and martensite maybe more at the surface and less at the interface or near the base metal. Similarly the grain size and shapes may also vary from the surface to the interface of the modified your zone.

So, for analysing the different sizes and shapes and their distribution as well as proportioning of the various phases and identification of the; and their distribution, the image analysis is carried out. So, for the image analysis of the microstructure image analysing software's are used. Images analysis one typical software which is available in public domain for the analysis purpose and this is the one which also can be used for the image analysis purpose so that micro structural analysis can be performed.

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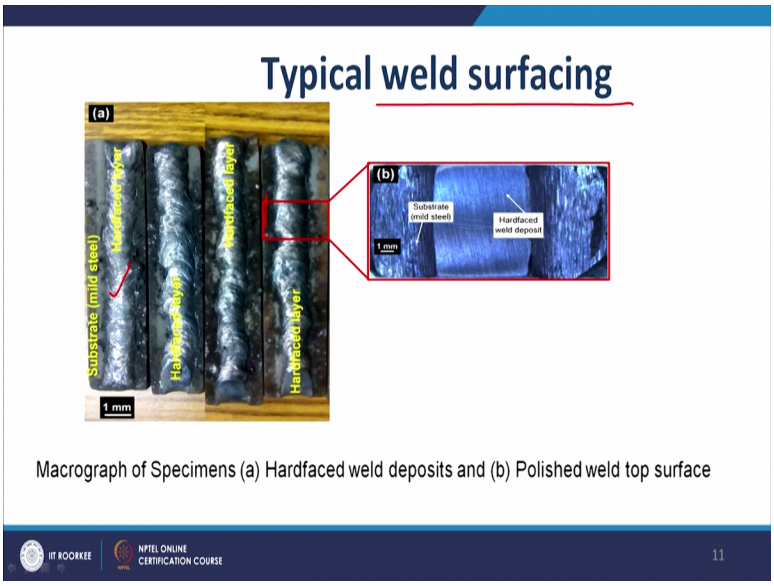

As far as the characterization metallurgical characterization is concerned will take up the example of the weld surfacing, so here the weld hard facing is on the mild steel substrate is carried out and we take to the sample from this zone and it analysis was performed. So, here both sides we have the substrate of the mild steel and in between there is weld surfacing deposit.

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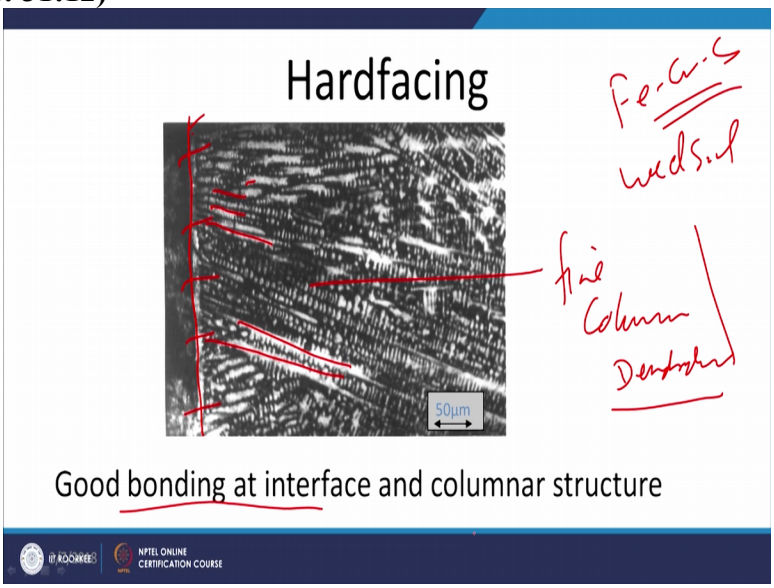

So, what it shows that at the interface of the base metal there is all these are the columnor dendratic grain. Primarily it shows the fine columnor dendratic structure in the weld surfacing of iron chromium carbon system. At the interface its sound there is no crack there is no porosity. So, the good bonding at the interface plus it is coupled with the kind of the columnor dendratic structures and now it can be used like since this is dendratic structures we can see the intra dendratic Rv spacing for sizing.

Since and this is a columnor dendratic structure spacing can be used. Here we can see some kind of the planar structure also is visible just at the interface. But there after it is primarily the columnor dendratic structure. So, now here I will the summarise this presentation, in this presentation basically about the need of the performing chemical analysis and various techniques which are used for performing the chemical analysis of the modified surfaces and what are the different features that we try to look into the micro structural aspects of the modified surfaces. Thank you for your attention.