

Heat Treatment and Surface Hardening (Part-II)

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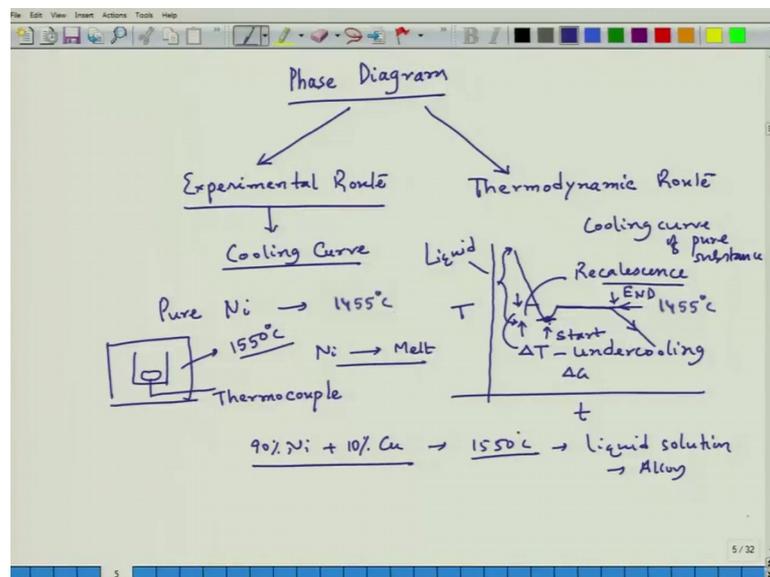
Indian Institute of Technology, Kanpur

Lecture - 04

Determination of Phase Diagram (Experimentally) – I

Hello everyone. Let us start forth lecture.

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And today we will talk about phase diagram. And here we will concentrate on binary phase diagram. And in the last lecture as we have mentioned that there are two ways one can find out phase diagram; one is experimental route and another one is thermodynamic route. And as we have mentioned that phase diagram is integrate part of heat treatment, because that initially that diagram gives you an idea that what sort of phases you would come across, if you start doing treatment at a particular temperature, and for certain duration of time; it gives you an idea that what would be the phases.

So, that is for, it is very important to know phase diagram of a system. So, first will start with experimental route, and in phase one, we talked about two phase diagrams; one was isomorphous, another one was eutectic. And today we will try to get isomorphous phase diagram as well as eutectic phase diagram, a simple eutectic phase diagram, after doing a couple of experiments of series of alloy systems, series of alloy mixtures. Now when we

do experimental route that time, the first thing that comes into our mind which is cooling curve, and let us talk little on cooling curve.

Let us say we have taken a pure metal, let us say pure nickel, whose melting point is 1455 degree Celsius. If we take nickel in a crucible, and then take it inside the furnace, and also we connect a thermocouple in order to see the temperature of this nickel, and then we take it, take the temperature of the furnace to around 1550 degree Celsius. Then all the nickel will melt. Now after that we can keep it inside the furnace and put up the furnace.

Then we will see that a temperature is gradually going down and the point when we are switching of the furnace. From that point onwards if we try to see temperature variation with time, then will get a plot like this, the temperature will gradually go down of this nickel piece, nickel melt as for the furnace is cool down, that particular situation you will get a plot like this, the temperature of that melt is gradually decreasing.

And as the temperature of the furnace is going down because you have switched it off, and it is cooling very slowly, so you will come across a kind of graph, like this graph, like this. Where, if we measure this particular temperature, we will see this is around 1455 degree Celsius, and there is a deep, and this deep in temperature we have also discussed before, this is called recalescence. This happens, because in order to get some solid crystal in a liquid melt, we have to cross interfacial energy barrier, we have discussed it before.

And that is what we have to, have a sufficient under cooling. This we can call it as ΔT which is under cooling. And during this under cooling, we have corresponding ΔG variation, and that gives free energy which is negative term. Actually supplies the energy for the formation of interface. And once the first nuclei forms here, the first nuclei forms, and then since the solid is forming, it has to eject the amount of latent heat, which is required for solidification.

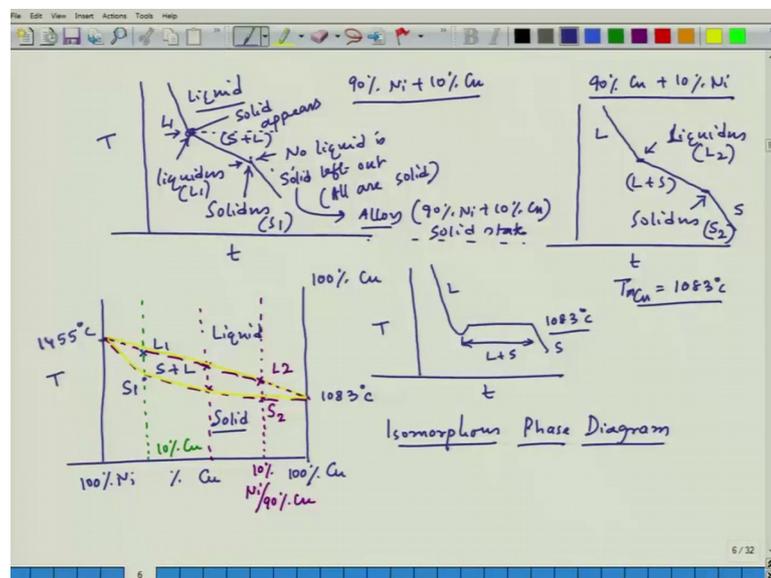
Since it is leaving that much of heat to the melt, the melt temperature again goes up. And that is what we have a deep and then going up. And finally, it reaches very close to the melting point rather it is little less than melting point, because at melting point nothing happens. And then it continues till all the liquid solidifies. And here we are not seeing

that regular arrival of this recalescence, because that time already you have crystal solid nuclei.

So, whatever heat you are taking out, and that is heat is nothing but equivalent to the latent heat of solidification. And then once you have all the liquid turning up into solid, then again temperature starts decreasing along this path. So, this is basically start of solidification, and this is end of solidification, and this is start. But actually its happening, if we see this particular plateau this is happening at around 1455 degree Celsius. So, this is a kind of cooling curve, we call it a cooling curve of pure metal pure substance.

Now, if we take an alloy, then situation differs. Let us say if I take 90 percent nickel, and 10 percent copper, and if we take the temperature to 1550 degree Celsius, then this will form an alloy, and since we know that alloy is nothing but a solution of two or more metals. Here also it will form a liquid solution, which is nothing but alloy of 10 percent copper in nickel. Now here, since nickel content is more so that said, we can say that it is a solid solution of nickel, it is an alloy of nickel rather where we have 10 percent copper.

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Now, this alloy we take then we try to do the same experiment, we will get a cooling curve again. So, now, as we understand that cooling curve is nothing to the temperature time plot, as the metal is cooled down from liquid phase. Now here initially it will cool down like this, where everything would be liquid. Here also if you see till this range, till

this range it was all liquid, and once recalescence happens then solid starts. Here also it will be all liquid, the liquid temperature goes down, but interestingly. Here the situation does not happen like this rather it goes like this at a slope, which is different than the initial slope.

And then again after some time, we see there is another, rather it would be, and this is 90 percent nickel plus 10 percent copper, and we can actually see that, at this point solid appears, and at this point no liquid is left out, means all are solid. So, then we can say that this point we call it as liquidus, and this point we call it as solidus, and here we have liquid here. We have complete solid, and this is nothing but an alloy of 90 percent nickel plus 10 percent copper in solid state, and in between from liquidus to solidus, we have a mixture of solid plus liquid.

We will come to this particular part later on, because this phase we are just indicating phases, but the composition varies continuously from this point to this point, this point to this point composition varies, and that variation of composition as well as the fraction of solid and liquid can also be determined. Once we get an idea of phase diagram, this is 90 percent nickel and 10 percent copper. Similarly if we make it 90 percent copper plus 10 percent nickel, it would also form solid as well as liquid, solid solution rather solid alloy as well as liquid alloy, and that time we call it as an alloy of copper, where 10 percent nickel is present. And in case of copper nickel system, we know that they can be mixed at any proportion in liquid as well as in solid state.

So, if we try to follow this particular alloy, and try to do the same experiment; that means, putting it in the furnace heating it at around of close to 1400 degrees Celsius, sorry here also you have to heat, it will around 1550 degree Celsius melt everything. Once you melt everything, then they will form an alloy, and then you start cooling down rather put up the furnace, and then start seeing the temperature variation. There also will see a similar plot, but if we see the same axis, this is temperature, this is time. If we follow the same axis, then we will see that the melting fast solid that appears would be little lower temperature than these I will come to that why it is that, and then here also, this point also would go down little bit, and then finally, you have a situation like this, this is also liquidus, and this is solidus, and here it is liquid here, it is solid and it is liquid plus solid.

Since majority is copper and copper melting point is 1083 degree celsius. So, that is what the entire temperature points for liquidus, and solidus. They drop down and finally, if we see 100 percent copper. Again we will see the similar plot, like what we have seen in case of 100 percent nickel. Again time temperature we see a plot like this, where the same thing happens. What happened in case of nickel, only thing is this time the temperature would be which is the melting point of copper melting point 1083 degree Celsius and here also we could see a recalescence.

In other cases also, in case of alloys also, we do see recalescence, but the effect is lesser than, effect is not that significant in compared to in comparison to pure copper or pure nickel. Now here also in this zone, in this zone I, see that liquid is gradually converting into solid. So, it is a mixture of liquid plus solid, and here it is liquid, and here it is solid, but in all those cases, those are pure substance, and only difference is in case of alloy, we have a slope, and we will come to know why that slope is. And here also we do not see any slope, it is a flat line which is horizontal with reference to time axis.

Since the single temperature all the solidification is taking place, but here we do not have a single temperature of a solidification is taking place, rather in range of temperature of a solidification is taking place, and the reason being, there is a continuous change in composition . So, now, if we see this one, this is pure nickel, and this is pure copper, and if we try to plot those solidus and liquidus points on a diagram, where the axis is temperature, and here the axis is percentage of copper.

So, here 100 percent nickel, and here 100 percent copper, since in case of copper and nickel we are getting a single temperature, where all the liquids are converting into solid. So, in case of copper, we can point out it is 1083 degree Celsius, and here it is 1455 degree celsius pure nickel melting point, and pure copper melting point. Now if we consider, as we have considered in case of these, this particular case, where 90 percent nickel and 10 percent copper.

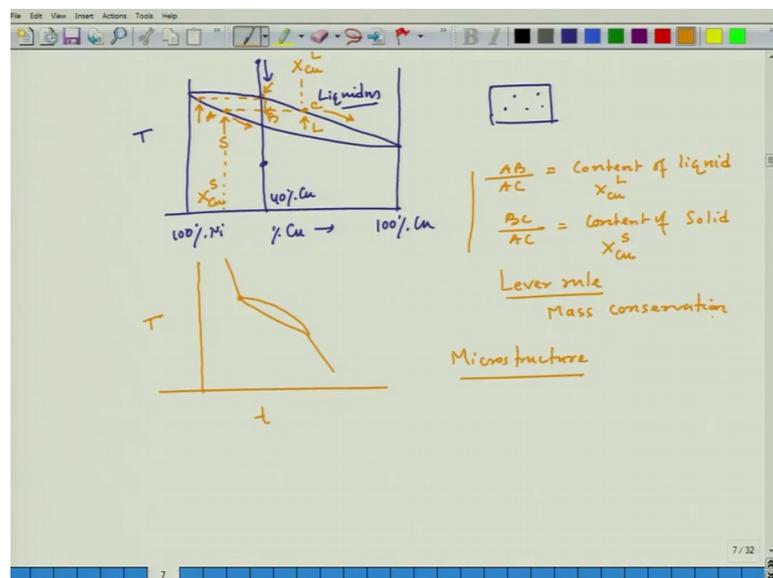
Now, I just put a dotted line, this is 10 percent copper, we see that this point, if we try to compare with this point, this will be this liquidus line is lower than the melting temperature. So, you just try to put that particular point, which is, I would say that, this is l 1, this is this is l 1, this is s 1, if I point at l 1 as a liquidus and solidus is a s 1, and here it is l 2, and this is s 2, then this is my l 1, and now the another temperature is s 1.

So, the same composition we are getting two points liquid line; liquidus point and solidus point. Now on the other side, if we try to compare, try to put this values here on the other side, this is. Let say this is 10 percent nickel or 90 percent copper, then there would be two points again, and those points will be lying lower than these points. So, this would be, here this is l 2 and here it is s 2.

Now, if I try to connect all the l 1 point, l 1 and l 2, and if we can also get some composition midway, we can also get corresponding l liquidus, and solidus lines points. Now I connect those, and gradually if we take it down to pure condition, and then I join the liquidus as well as solidus points, I get a plot like this, where the final plot becomes this. And so above L 1 everything is in liquid, and below s line everything is solid in between. As we have seen that is a mixture of solid and liquid, here also is a mixture of solid and liquid. So, we say that solid plus liquid.

Now, what we are getting is a phase diagram, which we call it as isomorphous phase diagram. And now let us look little more into the isomorphous phase diagram, because that is also crucial.

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Now, if we try to draw an isomorphous phase diagram, what we have seen in case of nickel copper, this is 100 percent nickel 100 percent copper and percentage at copper increases this way, this is temperature.

Now, let us take any compositions like this, and if we take any composition, let us say we are taken 40 percent copper, if we take the temperature to this level, everything would be liquid; that means, we can say it is a liquid alloy, and if we can take it to this, we can say that it is a solid alloy, but when it cool along this line first time, when it touches this point, which is basically liquidus line, where first solid appears. The first solid would appear here. Now if we have a melt like this, where some solid crystals are forming, then there will be compositional changes.

So, now once you have at the first crystal forms, first solid and that solid will have composition here, and the liquid composition would remain here, because its a tiny change. So, overall composition would not change the liquid. Composition would not change much, but the first tiny crystals would be having composition at this point. Now still we cool it down further, then if we cool it down further. So, if I reach to this temperature, we will see that there will be liquid phase, and there will be solid phase, and the liquid would have composition here, and solid would have composition here, this is solid, this is liquid.

Here it is basically nothing but the composition X copper solid, and this we can say that X copper liquid, and interestingly we can also calculate what is the mass fraction of both the phases liquid and solid. We can say that if we connect it as A B and C, we can say that $\frac{AB}{AC}$ by $\frac{BC}{AC}$, this is the fraction which is the content of liquid with A composition X copper liquid, and $\frac{BC}{AC}$ by $\frac{AB}{AC}$ equal to content of solid with a composition X copper solid, and this is coming from lever rule, which simply follows mass conservation.

Now, as we see that the composition is changing, as we are going down, mainly the liquid composition is changing. So, the liquid composition is here. Now when we cool it, we are getting a plot like this. Now interestingly at this point which indicate this point for solid appears as we are going down. Then we are changing the composition of the liquid as well as solid. The solid which would appear is basically varying along this line; that means, as well as the liquid, which would get solidified is also varying along this line. So, every time I am saying there is a lowering of solidification temperature.

So, that is what it takes a shape like this, though I am, we have drawn it like this. It may not be same as what it is a flat line. There could be a curve like this, because every time the amount of latent heat; that is also changing at the same time, heat extraction rate

would also change, depending on the condition of temperature at the surface. So, that is what you have a slope like this, and typical shape of cooling curve . So, now, later on we will also check what will be the microstructure, microstructure which is also important, how the microstructure evolves as we go down in that, along this particular any composition line in case of isomorphous system; that is also very important aspect, because finally, during heat treatment we are aiming at some particular microstructure.

So, how the microstructure evolves, that is also an important aspect, and will also consider this part later little later, but we have now seen that how experimentally one can determine a phase diagram. We have given example of isomorphous system in case of copper nickel. And now in the next lecture we try to see how an eutectic phase diagram be drawn from simple cooling rates, and that cooling rates can be found out cooling, not cooling rate this, rather cooling curve, and those cooling curves can be found out from a simple experiment.

So, let us stop here. Now in the next lecture we will try to get into eutectic phase diagram, and how to get eutectic phase diagram from cooling curve determination. Let us stop it.

Thank you.