Thermo-Mechanical and Thermo-Chemical Processes
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Lecture-25
Processing Maps: Case Study

Hello friends, continuing with our processing maps discussion so, as I promised you we will have a case study in the last lecture in this particular module of processing maps. So, the case study is based on our own work. So, already we have kind of seen that there is no agreement and final word on the model which can be used for generating a particular processing map, but for this particular work we have stick to the dynamic materials model.

And the idea here is, in this particular case study that it was felt that the when you are generating processing map, we are only using flow stress data which is we have obtained from hot compression test. So, you compress a cylindrical specimen, measure the flow stress, measure the strain, strain rate as you know that how fast you are pressing or applying the compression strain to the material, by that I know strain rate. I know the temperature and from flow stress, we are calculating the processing map.

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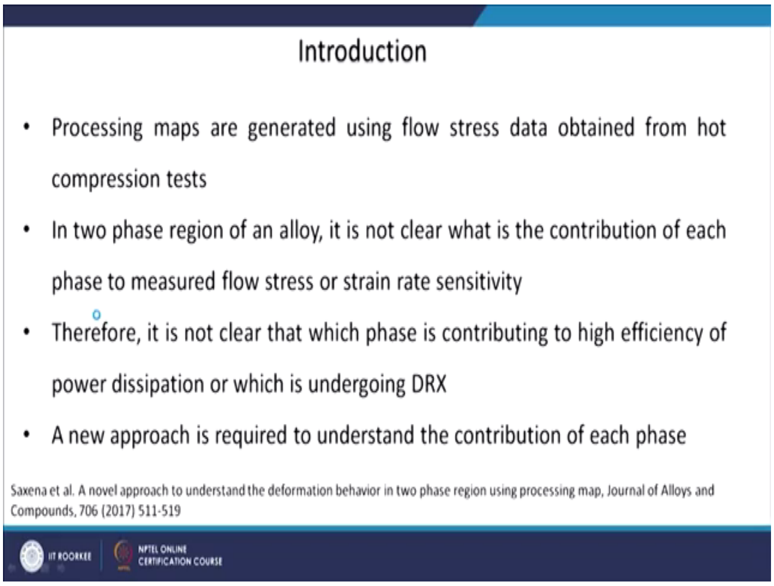

But the thing is that as already I have pointed you that the material behaviour is a very important factor in in getting all this or using all these models. And if any material is there which shows a phase transformation, simple example of steel you have ferrite phase which is at lower temperature, lower than 725°C. Above that you have 2 phase region, so α start to transform into

Austenite. And then at high temperature depending upon the carbon percentage in steel you have fully austenitic phase. Ferrite is a BCC phase that means body centred cubic is the crystal structure, Austenite has a face centre cubic crystal structure. So, you can understand that the deformation behaviour of these phases will be quite different but when we are developing a processing map, for example, steel sample we are deforming, let us say from 650°C to let us say, up to 900 or 950°C maybe at 7, 8 different temperatures. We are covering single phase α maybe couple of temperature then 2, 3 temperatures in two phase region, where α and γ both will be in equilibrium.

And then you are covering maybe couple of temperatures or two three temperatures in the austenite field and knowing that these phases will deform in very different fashion, as we told in case of constitutive equation also, that developing one single constitutive equation is not an accurate or a good idea for all the temperature range, because different phases are stable are in equilibrium at different temperature.

So, we propose that it should always be the phase or information about phase should always be there in the development of constitutive equation as well as processing map. So, when we are developing processing maps, we for example, in two phase region, if you are in single phase, it is not an issue, but if you are in two phase region, now if you have a two phase, we are not sure, if you are getting a high efficiency in these two phase region.

So, this high efficiency is due to dynamic recrystallization in α phase or let us say if you have α and β two phases so, whether the high frequency is due to dynamic recrystallization in α phase or β phase or which is contributing to the efficiency that we do not know? Or in that particular deformation zone, two phase region whether α phase is the dominant phase or β phase is the dominant phase?

Already Zirconium and Niobium alloy in our work on constitutive equation we have seen that from activation energy calculation that α phase is the dominant phase. So we wanted to see whether we can see it in the processing map also. So to understand the behaviour of these two phases in a two phase region, we should have some new input to the processing map.

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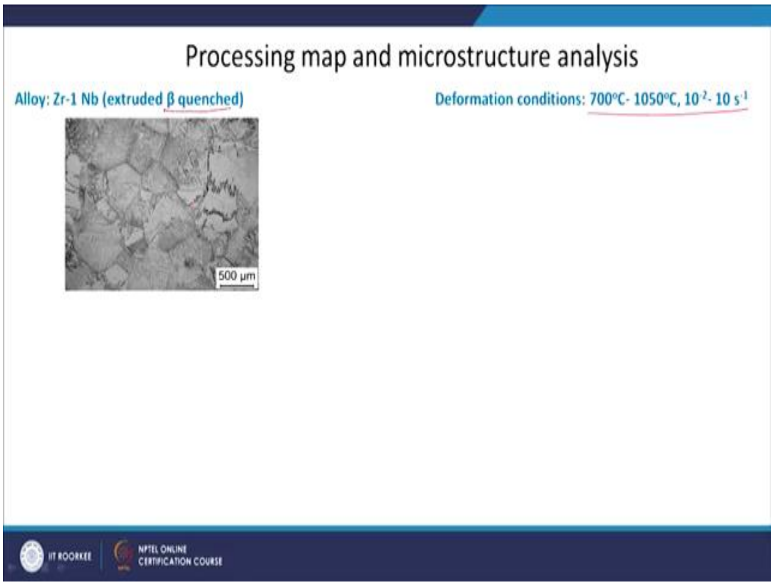

So for the work, Zirconium and Niobium alloy was taken and the deformation conditions were these, 700 to 1050°C, the initial microstructure was like this extruded β quenched, equiaxed microstructure with whatever you are seeing this (refer to above figure) needle shape is the martensitic transformation because of β quenching. So β to α transformations is through martensitic transformation. And that is why you see all these kind of needle shape microstructure.

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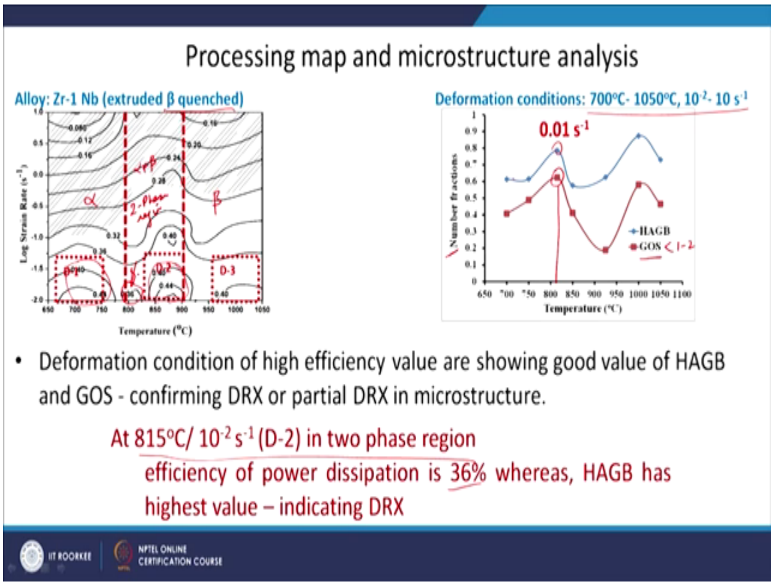

So this was the processing map (refer to above figure) and you can see that these are my high efficiency regions and when we did the microstructural analysis, as I have already mentioned about parameter called Grain orientation spread that tells you about whether a grain is recrystallized or not. If it is recrystallized then, the grain orientation spread value should be low.

So, here I am not plotting the grain orientation spread value what I am plotting is the fraction. So, we are plotting the grains which has a grain orientation spread less than if I am not wrong the criteria was taken as I think one or two. So grain orientation spread was taken as some value that it should be less than one and how many fraction of grains have this value.

So, if fraction is high, that means the microstructure is recrystallized, if friction is low, microstructure is not recrystallized. So when I see a high value it should be recrystallized. So what I am seeing from grain orientation spread here, that you are seeing highest value at a temperature of around 815°C and then it is coming down and again some high value at 1000°C.

And similarly the high angle grain boundary fraction, so in dynamic recrystallization I can expect that the fraction of high angle grain boundary will be more. So that is again showing the same trend and showing a very high value of 80% at this particular condition. But when you see on the processing map at this particular condition my efficiency is not that high. It is only 36% whereas, when these values are started coming down there you are seeing more efficiency 44%, similarly here (refer to above figure).

So, somehow we are feeling that where we have actually two phase region so, this is the two phase region of this particular alloy (refer to above figure). This is my two phase region. So, I have α phase here, β phase here and α + β here. So, wherever this phase transition is taking place, there this anomaly is taking place that I am not getting the high efficiency. And these are the three domains identified of high efficiency from the processing map (refer to above figure).

So, after that, this is what I have already told you deformation condition of high efficiency value are showing good values of HAGB and high friction of grain orientation spread confirming DRX or partial DRX in microstructure, whereas in this particular condition which is in two phase region the efficiency is low 36% though the DRX percentage and expected DRX is very high.

So, it should have shown high efficiency value in this particular condition which is from our microstructure we are expecting that but it is not giving that.

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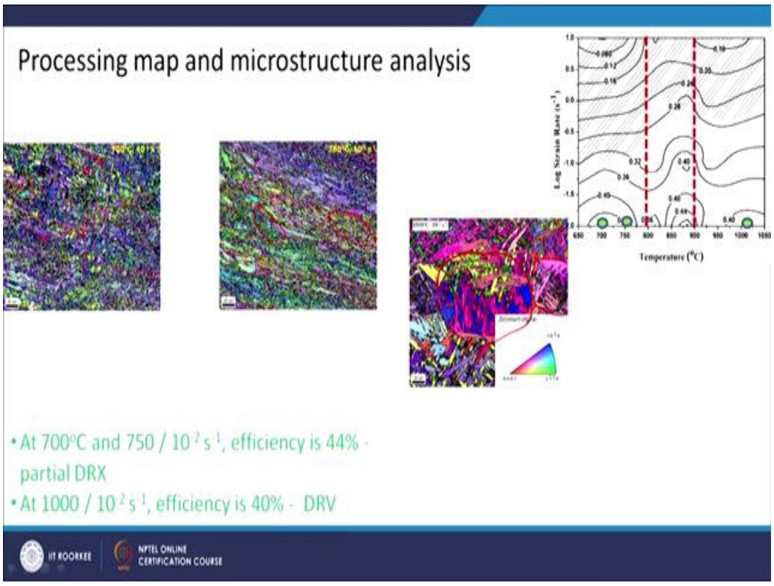

Some more microstructural analysis at different conditions (refer to above figure). For example, at 700°C you can start seeing some recrystallized microstructure, lot of fine grains, but also these kind of elongated grains. You can see lot of elongated grains here. So some big elongated grains are there and some very fine recrystallized microstructure can also be seen here and this is where you have high efficiency.

So, it is partial dynamic recrystallization can be seen here. Another high efficiency zone at 750°C at 10^{-2} strain rate, again you can see some elongated grains and also some very fine grains in between them. A lot of these fine grains are there. And these elongated grains are also their big coarse grain. So, again some grains have undergone recrystallization, partial recrystallization is there and some grain are still under the deformed state.

Or maybe some recovery has taken place but it is again confirming our high efficiency in this particular deformation condition. This is a microstructure in the β phase field at around 1000°C or so. And you can see that of course, this is also a recrystallized microstructure but after recrystallization grain growth has taken place. So, in our previous lecture also we have seen that when we go to lower strain rate higher temperature the grain growth takes place.

So, this is what actually this if I want to tell you this is a one grain. Similarly, this is one grain here and the plates you are seeing is because of the martensitic transformation. So, the material was quenched after deformation at 1000°C and from there it is quenched. So, all this this kind of plates you see these are all because of martensitic transformation, and martensitic transformation can takes place at different these are different variants (refer to above figure).

You do not have to worry about that, but it composed of one grain. So, this is a very big grain maybe around 100 microns or 150 micron or so, whereas these are very fine microstructure. So this is also a recrystallized microstructure, but also have seen considerable grain growth in the β phase field. So, at 700-750°C, 10^{-2} efficiency is 44% and also we are able to confirm it from the microstructure that either partial DRX or dynamic recovery is there.

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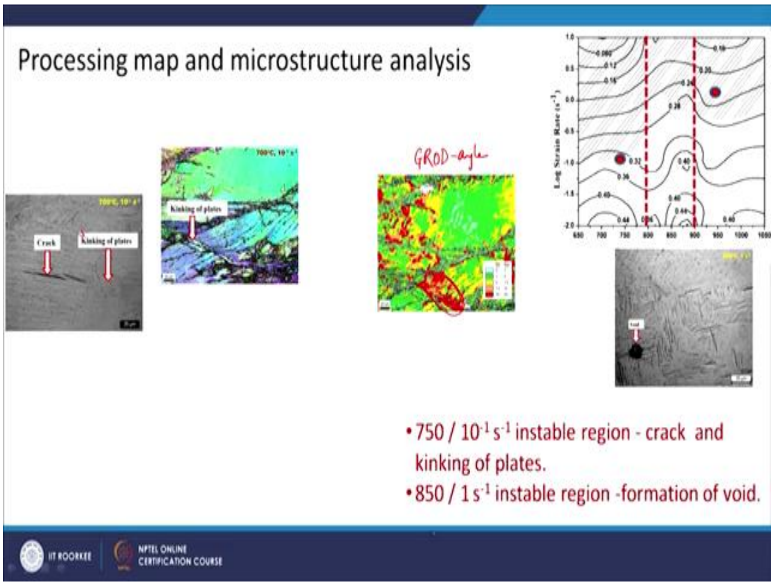

So now we are looking at the instable regions. So this is one of the condition where we were seeing the instable condition and we can see at that particular condition, we have some crack formation is there. And also one instable microstructure I told you earlier that it is called as kinking. So two plates are there and they got kinked. So kinking is there at 700°C at 10^{-1} and we kind of did some more detailed study about this kinking that how this kinking takes place.

So, this is what is a kink, you can see these α plates (refer to above figure). All these are α plates and they got kinked at a particular location. So this is a single grain and in that you have α plates and there is a kinking. So this we studied again these are all our EBSD analysis. The microstructure is through EBSD analysis only. So we have plotted another map which is called GROD Grain Reference Orientation Deviation.

And also there is an angle. So that means it shows that how the grain is rotating due to the deformation. So, there is a reference orientation and how much it is deviating from that. So, you can see that all the red colours means the deviation is more. So, this kinking how it is taking place within the grain is that due to deformation, the grain is rotating because of the when the dislocation traverse through the grain actually it rotates the grain.

That is how you get the low angle boundary and then high angle grain boundary. So, it is rotating the grain and when the grain is not able to take that strain, so there is a kind of a kinking of the plate or kinking of the grain. So, that strain which is being developed in the grain, getting relieved through this kinking. So, if you see in this particular grain which we have shown here that after this the very high value of GROD the other side of the kink, you again see it is it has a very low value.

That means all the strain or because of that whatever strain energy which was developing that got released through this kinking process and on the other side you do not see any development or any high value. So, on the other side it got relieved through this kinking process. So, this deformation and the accumulation of deformation within the grain creates this and give rise to this kinking, that is what we proposed (refer to above figure).

Then there are some more defects for example, a void formation at a higher temperature. So, from there we could see that these are the instable regions 750°C 10^{-1}, 850°C 1 and we could see that there are some defects present on those conditions also.

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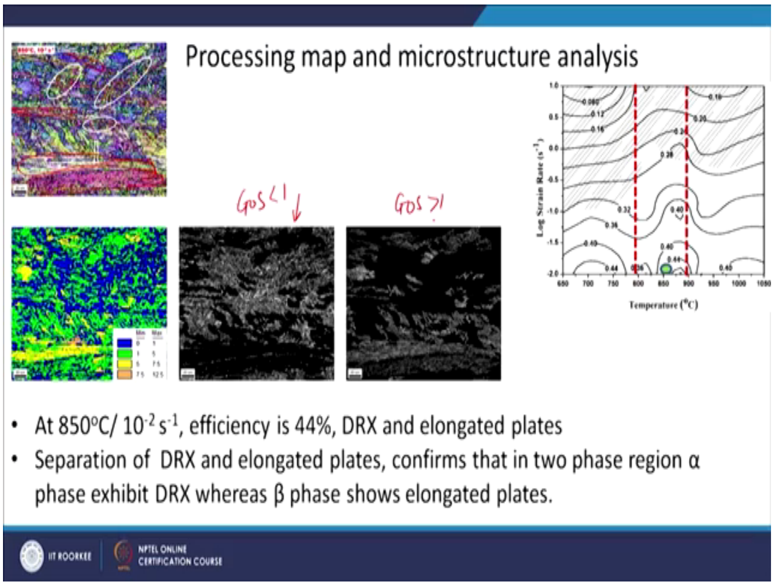

So, now looking at another area of where we had high efficiency that is in the two phase region now. We are coming in two phase region now, till now we were looking only the single phase. And now we see some interesting thing here. Though it shows very high efficiency so, I can see some recrystallized grain which are shown this with oval shape (refer to above figure). But I also can see that there are some big elongated grains, these are all big elongated grains.

Some grains are here also (refer to above figure). And in between them there are lot of these fine recrystallized microstructures are there. So, we wanted to know that whether any particular phase is responsible for this kind of behaviour. The only problem is that in these kind of alloys when you do cooling, everything again gets transformed to one phase. So, if you are in two phase region α and β, maybe α is doing something or β is doing something.

But you cannot preserve what β was doing, what α was doing because when you do the cooling, both will be have α phase only. The both will look very similar. To segregate that, of course, we also plotted the grain orientation spread map. So, you can see all these fine grain regions are showing low value of grow grain orientation spread. That means these are recrystallized grains and all these big ones are showing higher value.

That means they have not undergone recrystallization and based on this GOS we have also separated the two type of grains. So, this (refer to above figure) is the one which has a lower GOS. GOS is let us say less than one and this is GOS more than one. So, you can see two different clear microstructures here. So, from here actually we also tried to figure out whether this is the elongated one, is the initial β grain and these are the α transformed, the α grains which got recrystallized.

That we kind of proved by showing that these particular grains have some transformed plates. During the transformation, they will have some kind of martensitic transformation whereas α because already it is α it cannot have a transformation. So, these grains are not showing any plate type of formation, but these all big grains are showing formation of martensitic plates.

So, through that you can say that these big grains are from β grains and all these very fine recrystallized grains are from α phase.

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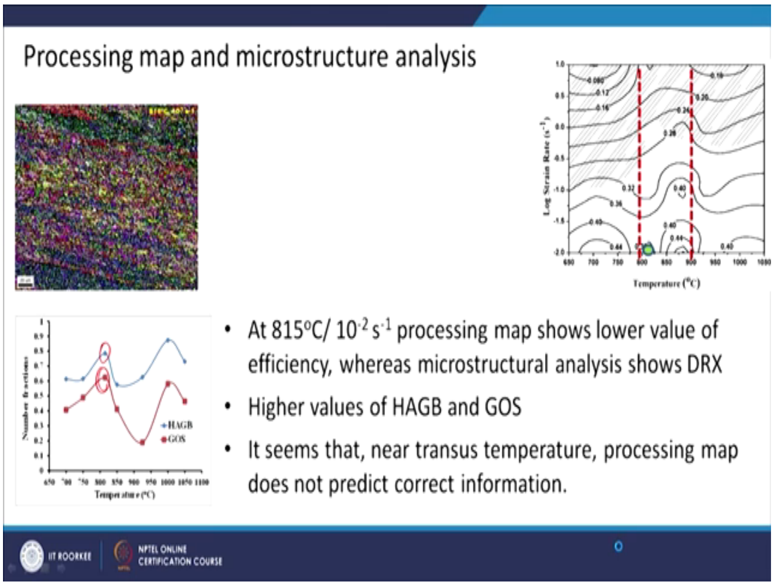

And to confirm our earlier analysis that at 815°C though it is showing lower efficiency, but in microstructure, I was seeing high fraction of grains with low grain orientation spread. Similarly, higher friction of high angle grain boundary. And this is the microstructure very different from the earlier microstructure. Now, you can see very large number of and the whole microstructure is covered with very fine grains (refer to above figure).

So this is now fully recrystallized. And that is why you get all these microstructural parameter with these values. But in my map I do not see an high efficiency region.

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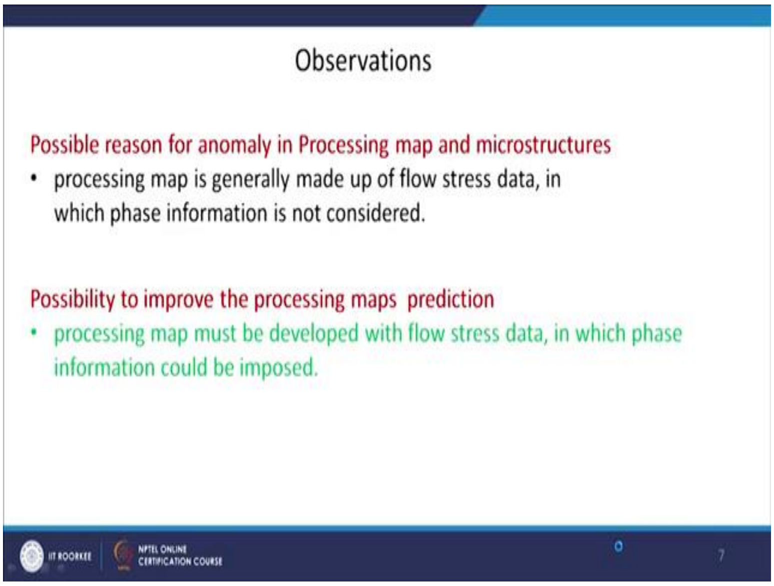

So, to kind of clear this or to find out the reason for anomaly, we felt that the phase information should also be taken as input in creation of this processing map. But, how to do that, that how I can put the phase information in the in the development of processing map? So, this is the method to calculate the flow stress data in the two phase region. What is the method? The flow stress of single phase is extrapolated in two phase regions.

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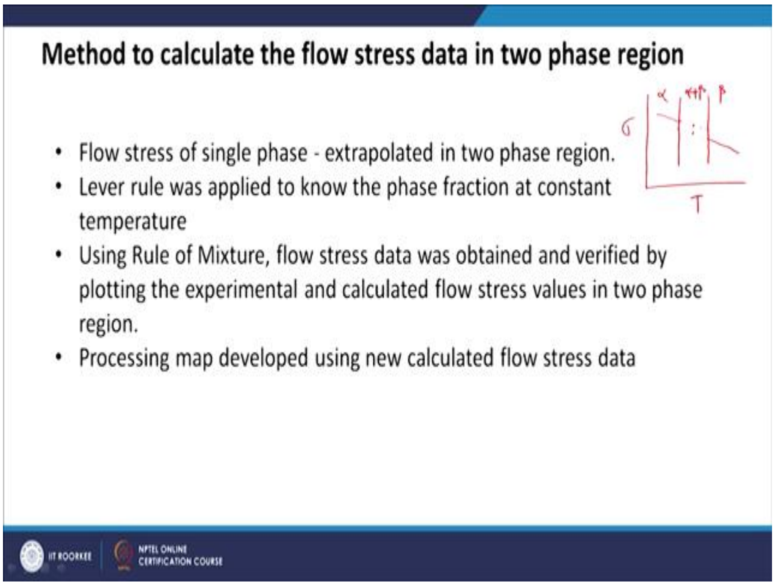

This is what we did in the constitutive equation also the case study where we developed constitutive equation. So, you have low stress data. So, this is my flow stress, this is my temperature (refer to above figure). So this is α, this is α + β and this is β. So, I have some flow stress data in α phase that I am extrapolating in the two phase region. Suppose I have some flow stress data in β phase that is I am extrapolating in two phase region.

And then I am using a rule of mixture here to find out what will be the stress of a composite of α and β. So, that is simply that what is the fraction of α phase multiplied by stress of α phase, what is the fraction of β phase multiplied by the stress of β phase and so, it is kind of a weighted average. Weighted average of the two phases and then we develop the processing map using this new calculated stress. So, this is how the calculation was done.

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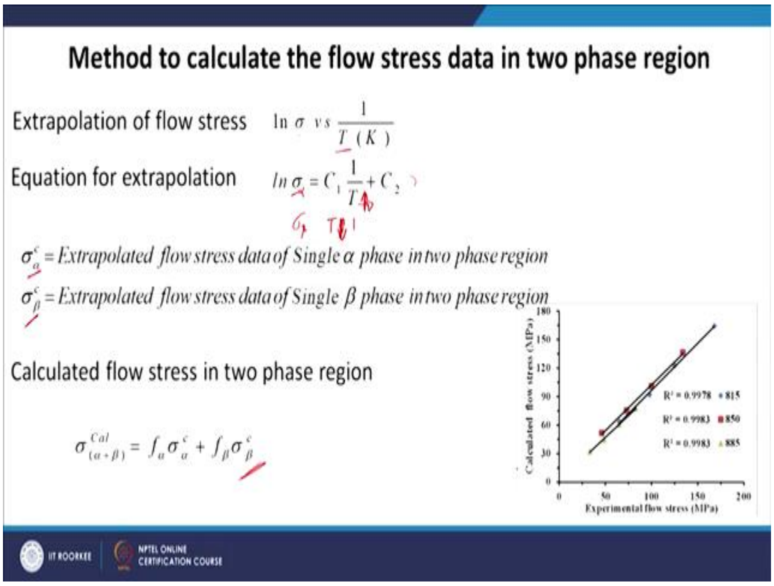

Already we know that σ relates with temperature in an exponential way, that is why we have taken logarithmic here. And I can turn that into a straight line equation. So, I can find out these constants. So, from there I can do an extrapolation that as I am increasing the temperature what will be the value of σ_α. For example, if I am reducing the temperature, I will have value of σ_β.

So, temperature is reducing in this case temperature has to increase. And from there I can have σ_α^C and σ_β^C. These are extrapolated values in the two phase region. And then we applied a simple rule of mixture what is the fraction of α. So fraction you can calculate from the lever rule of α and β. What is the fraction of α stress value of α which is calculated in the two phase region? And that will give you the calculated flow stress of combined α and β. So, these (refer to above figure) are the experimental flow stress and calculated flow stress just to see whether we are not very off from the actual flow stress values. Of course, the scales are different here. So, please be careful this is not at 45°. So, these are not exactly same stress and it has no meaning.

Then we should get the same processing map as what we are getting from the experimental values. So, these are the processing maps (refer to below figure).

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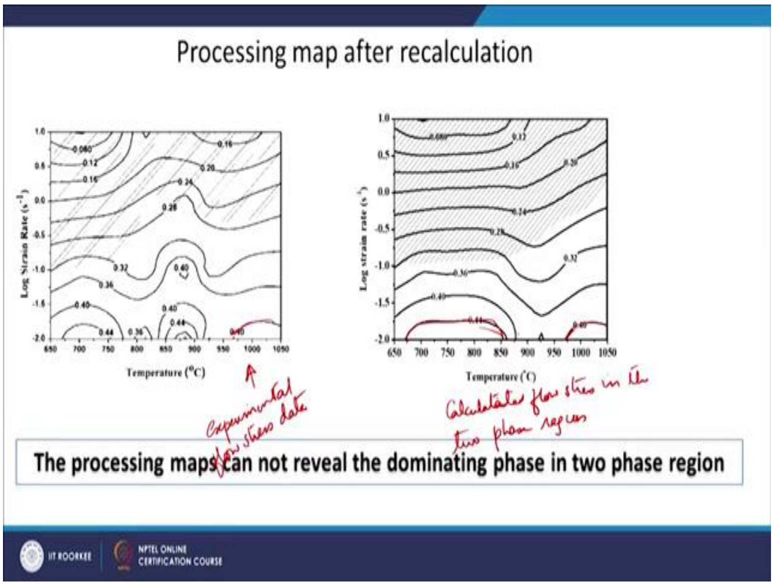

So this is our initial processing map from the experimental data and this is the map which we have developed after doing the calculation or calculated flow stress in the two phase region. So, single phase region we have taken the experimental values only. But in the two phase region we have taken the calculated stress value and now you can see the flow stress, the processing map looks quite different from the earlier one.

The main thing is that you are able to get a very high efficiency zone directly from around 675°C up to 850°C where actually we see either partial recrystallization or recrystallization. And in fact, at higher temperatures in the microstructure, there were elongated grains and only few grains were showing the recrystallized behaviour.

So that you are able to see that those location (refer to above figure) I am not seeing high efficiency. Of course, in the β phase field, the high efficiency same as what you get here because this is for a single phase. So, the main thing here is that we are able to get a high efficiency region for all the temperatures where we were able to see recovery and recrystallization. So, this is one confirmation, but the still the problem is that I still do not know from the processing map whether the recrystallization is taking place in the α phase or β phase.

Still it is able to show that the where the recrystallization is there in the microstructure, I should get my efficiency as high, but which one is the dominant phase? So for that, we did a simple thing that we plotted.

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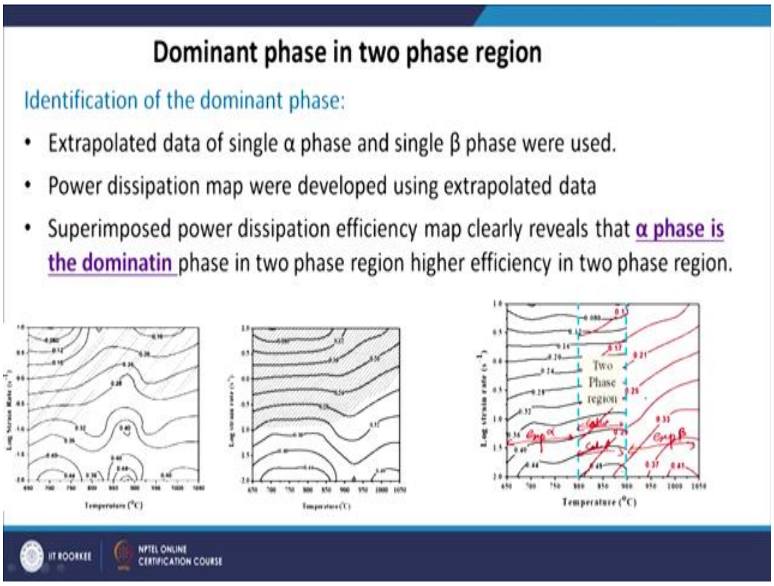

The extrapolated already we had stress for α and β phase separate. And so, we just plotted all the flow stress value of α which were experimentally found up to this temperature and here what we have found out from calculation for α. Similarly, these were the experimental values of β, this is for α, and these are the values for calculated β (refer to above figure).

And what we are seeing, you should be able to appreciate here that all these black lines are the efficiency contours for α phase. So, an α phase you have high efficiency of 0.48, 0.44 in the two phase region. Whereas, β phase if you see the highest efficiency is 0.33 at higher temperature. If you see in complete two phase region, it is around 0.29 or 0.25. So, the efficiency of α phase is almost double the efficiency of β phase.

That means whatever efficiency you are getting in the two phase region is because of the dynamic recrystallization in the α phase, not in the β phase. And that is contributing to the efficiency of high efficiency, which is you are seeing in the processing map. So, by doing this kind of calculation, where we have incorporated the information of phases, we are able to say that, I am able to capture all the high efficiency region, in the processing map, which is the usually the problem where you have phase transition and also which one will be the dominant phase, through this analysis. So this was our case study with our work. And I think you, you must have enjoyed this. So thank you for your attention.

Keywords – Processing map, Zirconium-Niobium alloy, Two phase region, instability.