Advanced Materials and Processes
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Lecture – 32
Superalloys (Contd.)

Welcome to NPTEL: myself Dr. Jayanta Das from Department of Metallurgical and Materials Engineering, IIT, Kharagpur. I will be teaching you Advanced Materials and Processes. We have just started our discussion on super alloys which is one of the best performing high temperature alloy. Compared to several intermetallics like aluminides, silicides, and let us say the high temperature martensitic steels.

So, superalloys has already shown their great potential and also this alloys are already used in gas turbine engine; where the performance of all these material is assessed in terms of turbine entry temperature. Because in this turbine section, the blades are required where stress, temperature and degradation in terms of oxidation are important properties to consider. And even though, several materials have been discussed so far, all this material and their performance has been summarized by considering this Larson Miller approach to rank the creep performance. During service life the performance of the material alloys has to be analyzed.

Now, today we will start the discussion on nickel base super alloy. However, among this whole periodic table why nickel is chosen as a base element that we will try to justify, because any alloy development required finding a base element, and choosing some other elements in order to enhance the properties like strengthening by introducing required phases. Or let us say to protect the alloy from high temperature oxidation. And so far, we have learned that aluminium, chromium and silicon these are the 3 element that provide protection.

Because aluminium from alumina, chromium from chromia, and silicon from silicon dioxide. So, let us try to start our discussion with this topic that, why nickel is used as a solvent in this high temperature alloys which is the topic of interest.

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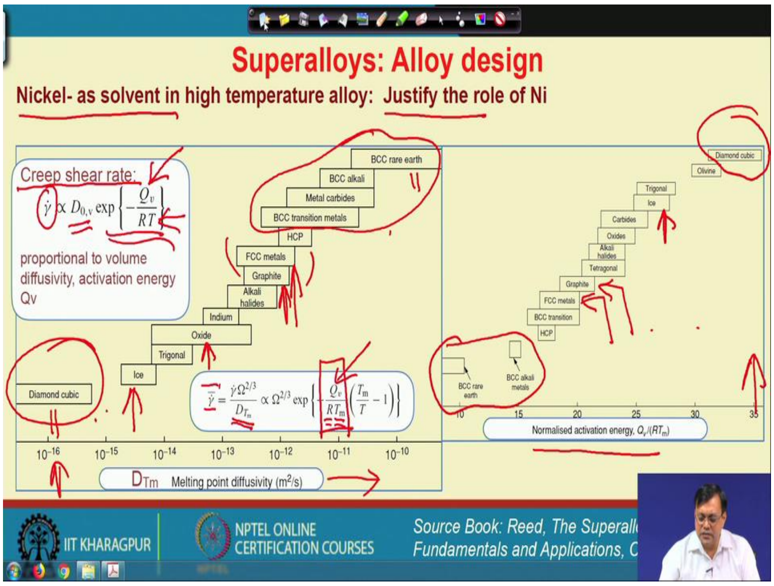

Superalloys: Alloy design

Nickel- as solvent in high temperature alloy: Justify the role of Ni

Creep shear rate:

$\dot{\gamma} \propto D_{0,v} \exp\left\{-\frac{Q_v}{RT}\right\}$

proportional to volume diffusivity, activation energy Qv

$\bar{\dot{\gamma}} = \frac{\dot{\gamma}\Omega^{2/3}}{D_{T_m}} \propto \Omega^{2/3} \exp\left\{-\frac{Q_v}{RT_m}\left(\frac{T_m}{T} - 1\right)\right\}$

Source Book: Reed, The Superall

Fundamentals and Applications, C

And we need to understand or justify the role of nickel. Since creep has been discussed a little bit regarding the high temperature performance of aluminide and nickel containing alloys. And also some sort of thoria dispersed tungsten.

So, the creep shear rate that is simply expressed in terms of the $\dot{\gamma}$, that is rate and with a constant and their exponential. This is basically very simple Arrhenius type of equation that we already know. So, basically this creep shear rate is proportional, it shows some proportional relationship with the volume diffusivity and the activation energy.

$$\dot{\gamma} \propto D_{0,v}\, exp\left\{-\frac{Q_v}{RT}\right\}$$

Here the activation energy is expressed in terms of Q_v. This is the diffusivity, the activation energy, and R and temperature. And this means that we can take a very different types of metals or elements. Let us say BCC elements, FCC we can categories in terms of their structure, because there have different voids and that void are basically the pathway for diffusion of those other alloying element. And on the other hand, the self-diffusivity has to be also considered. And we already know that the diffusion required for creep and the cell diffusivity have some close link.

So, here if we need to normalized the creep shear rate, then we have to consider the melting temperature of an element. So, here we divide this parameter by melting temperature and the D_{Tm} stands for the melt point diffusivity of a material. And so, before going to this equation, a plot is shown with melt point diffusivity (m^2/s) with different materials. So, the melt point diffusivity for a diamond cubic structure is very less than the BCC rare earth. So, you can see that the BCC transition metals and rare earth, all are lying here, whereas, ice has a relatively lower diffusivity than the BCC elements.

Now, what are the competitors? so, in between we find a FCC metal, or graphite. Now oxides are here. So, it is for sure that oxide can be a better for high temperature use, but since the toughness is very less and formability is also less. Hence, the FCC could be a better alternative. And now, if we take in a consideration of this particular term, that is the activation energy divided by R into melting temperature.

$$\dot{\gamma} = \frac{\dot{\gamma}\Omega^{\frac{2}{3}}}{D_{T_m}} \propto \Omega^{\frac{2}{3}} D_{0,v}\, exp\left\{-\frac{Q_v}{RT_m}\left(\frac{T}{T_m} - 1\right)\right\}$$

Then we normalize all these different class of elements and different structures and materials, then we see that the activation energy for ice is higher. Because this is just a reverse plot from the melt point diffusivity to the activation energy. The diamond cubic has the lowest melt point diffusivity, which is lying here with the highest normalized activation energy. Whereas, the BCC metals are lying here, why we are always talking about BCC and FCC? We will learn that in next few minutes. And here the FCC metals and graphite are lying.

So, FCC is somewhat better in terms of higher temperature application compared to BCC or other elements. Now, let us try to look at some of the other aspect, because for high temperature not only the melt point diffusivity and activation energy for diffusions are important. but, what about ductile brittle transition? Means I can choose a base metal element either BCC or FCC and it should be able to retain it is strength and plasticity, in terms of avoiding some sort of catastrophic failure throughout operating temperature use. And let us say for a turbine it will be cool down. Sometimes in room temperature again it will be used.

Let us discuss about flight. Everything is shut down when flight lands and then again we start a flight by turning on the gas turbine engine. So, there is always a thermal fatigue. Not only that there will be also a mechanical fatigue. Hence, properties should be consistent with the temperature, not only at the highest temperature, but throughout the working temperature range, which is very important aspect. Therefore, we a need to consider that.

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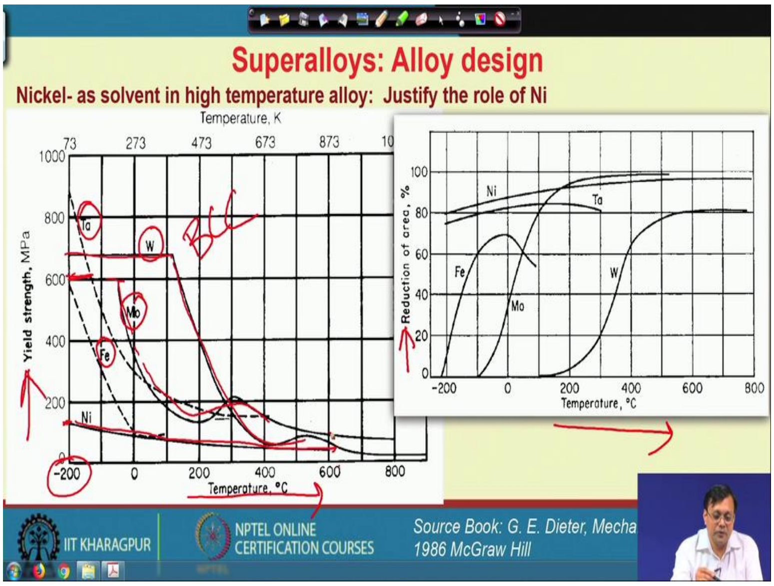

Superalloys: Alloy design

Nickel- as solvent in high temperature alloy: Justify the role of Ni

Source Book: G. E. Dieter, Mecha
1986 McGraw Hill

And let us have a look at a very basic mechanical metallurgy concept that we have learned quite a long time ago in the undergraduates. So, here some of the metals and their yield strength and reduction of area which is basically, an estimation of the plasticity, malleability, or let us say the ductility with the yields strength and the temperature is shown.

So, different element has their different melting temperature. However, if we simply consider a temperature and its strength means I am talking about a strength that is zero-time strength actually, means the yield strength. So, here nickel may have a low yield strength. However, it maintains the strength, even up to 600 °C. Whereas, if we think about molybdenum, tungsten, tantalum and iron. You see there is some common thing in between them that is basically the BCC element.

So, this element at room temperature or at -200 °C exhibit a very high strength; however, after just passing the room temperature, it falls or let us say tungsten has a very high strength, but it simply falls with the temperature. Now let us compare very similar situation in terms of the ductility.

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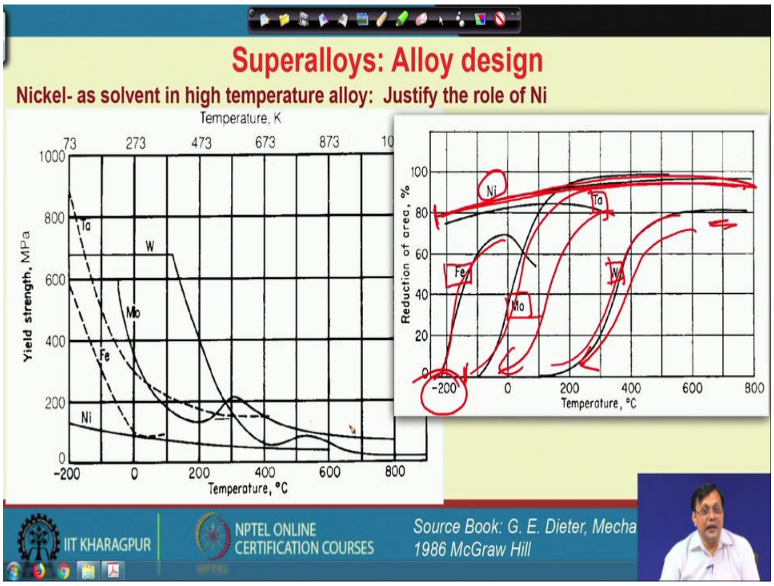

Superalloys: Alloy design

Nickel- as solvent in high temperature alloy: Justify the role of Ni

Source Book: G. E. Dieter, Mecha
1986 McGraw Hill

So, here the nickel is lying here. And all the BCC elements are here like tungsten tantalum. You see most of the BCC elements shows such kind of behavior.

So, even in a low temperature, these BCC elements simply losses its plasticity. So, if at higher temperature they are good, but it simply comes down. Whereas, nickel has a very wide range of very similar reduction in the area throughout the temperature range from -200°C; where a plane has to land in a polar region or to a very high temperature when it will be under operation. So, throughout this range it has a very similar property. And this is very important aspect to choose a FCC metal for this particular high temperature solvent atom.

Now, so far we have only talked about FCC metal. And why a FCC should be chosen for high temperature? Is there any other aspect?

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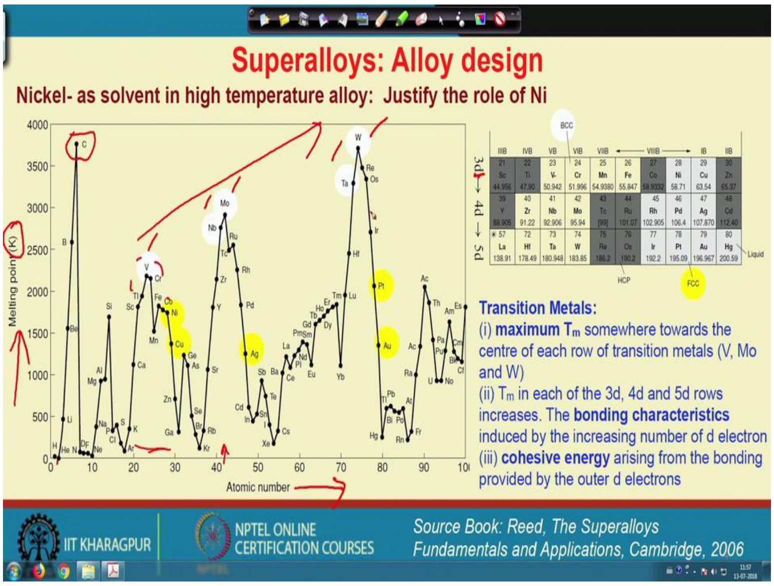

Superalloys: Alloy design

Nickel- as solvent in high temperature alloy: Justify the role of Ni

IIIB	IVB	VB	VIB	VIIB	VIIIB	VIIIB	VIIIB	IB	IIB
21 Sc 44.956	22 Ti 47.90	23 V 50.942	24 Cr 51.996	25 Mn 54.9380	26 Fe 55.847	27 Co 58.9332	28 Ni 58.71	29 Cu 63.54	30 Zn 65.37
39 Y 88.905	40 Zr 91.22	41 Nb 92.906	42 Mo 95.94	43 Tc [99]	44 Ru 101.07	45 Rh 102.905	46 Pd 106.4	47 Ag 107.870	48 Cd 112.40
* 57 La 138.91	72 Hf 178.49	73 Ta 180.948	74 W 183.85	75 Re 186.2	76 Os 190.2	77 Ir 192.2	78 Pt 195.09	79 Au 196.967	80 Hg 200.59

Transition Metals:

(i) **maximum T_m** somewhere towards the centre of each row of transition metals (V, Mo and W)

(ii) T_m in each of the 3d, 4d and 5d rows increases. The **bonding characteristics** induced by the increasing number of d electron

(iii) **cohesive energy** arising from the bonding provided by the outer d electrons

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Source Book: Reed, The Superalloys Fundamentals and Applications, Cambridge, 2006

Yes, let us see the whole periodic table of elements. And very interestingly I just plotted here the atomic number with the melting temperature, instead of melting point I preferred melting temperature in Kelvin. So, starting from hydrogen, you see there is a large peak where carbon is there. And now you understand why carbon is often chosen for high temperature material; this is one of the purpose. Now let us come to a higher atomic number site. So, there are some peaks here. And you see these peaks content vanadium, molybdenum, niobium, tungsten, tantalum.

So, what is common? these are all BCC elements. So, BCC has a lower activation energy required for diffusion. On the other hand, BCC has higher melting temperature. So, which one should be chosen or which one should be given more preference for high temperature material. I also told that a BCC element this molybdenum tungsten, if you exposed to higher temperature, in presence of oxygen they all produces volatile oxides.

So, volatile oxides cannot give you any protection from oxidation. And then we must consider some other things; however, it is for sure that this vanadium and these elements are lying in this 3d element. You see titanium, vanadium, chromium, manganese, and iron. So, they all are lying somewhere here. So, here is the vanadium and chromium; so titanium, vanadium, chromium, iron, cobalt. So, this is 3 and this is the 4d. Here let us say zirconium, niobium, molybdenum. And if you go towards this side actually, the d orbital electrons are increasing. And a d orbital electron signifies actually the bonding characteristic. So, like from here if you go towards the side the melting temperature actually increasing.

This is the symbol that if you go to this side then automatically the bonding become stronger. Or let us say, the cohesive energy is higher that is for sure. So, on the other hand, the maximum melting temperature is somewhat at the center of these each rows. So, like here we have vanadium.

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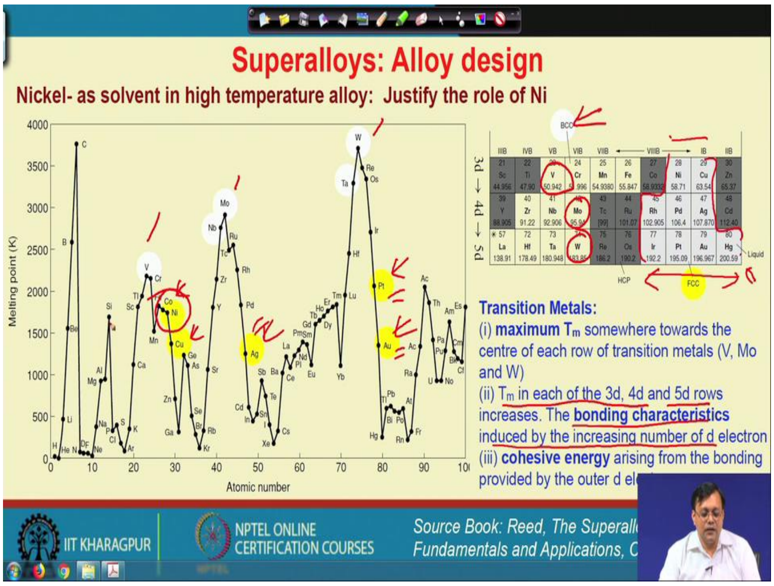

Superalloys: Alloy design

Nickel- as solvent in high temperature alloy: Justify the role of Ni

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Source Book: Reed, The Superall Fundamentals and Applications, C

And here we have molybdenum and we have tungsten.

So, these elements are lying here. Now, these I am unfiled blocks, these are all BCC elements, whereas, the slightly gray region is the FCC element; and from this theory if we consider it as a theory actually. So, probably these FCC metals may be better to choose. But you see these are all platinum group of metals. Means, platinum is very expensive, we cannot use it. A gold is also very expensive. So, for any industrial use it is not preferred to use actually; however, platinum heating elements are often used or also platinum sensors are used. So, where we have no other alternative, but here marker use a liquid.

So, there is no use of it for this high temperature metal. Now the melting temperature in each of these 3-d, 4-d, and 5-d rows it also increases. So, the bonding characteristic are induced by increasing the number of the d-orbital electrons. So, we have only choice in this particular series. So, here we see that nickel is one of the element, palladium is also very rarely used. And silver is also in that platinum group; which are quite expensive element.

And now, here the FCC elements are lying, platinum have a relatively higher melting temperature than nickel, but as far as I said that these elements are not preferred for any industrial purpose. So, we have only one alternative which is a nickel

So, because nickel has a 3d 8 or 9 electrons, it can be chosen as a solvent atom.

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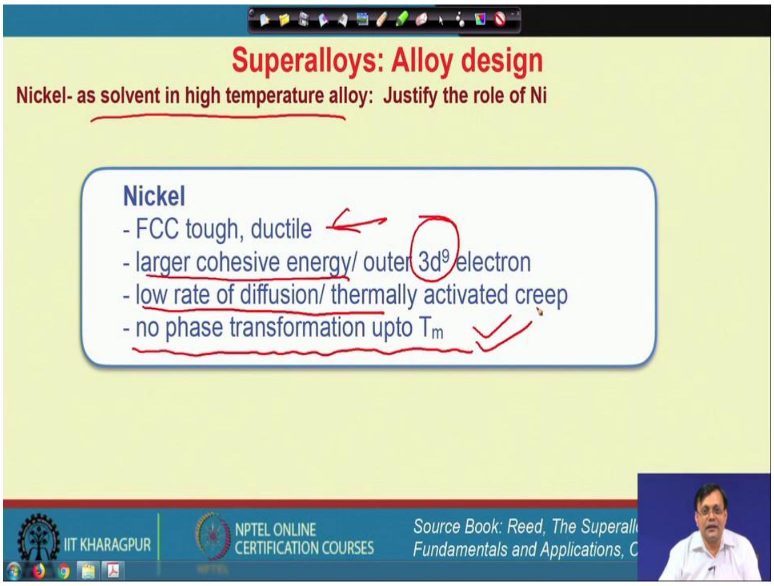

Superalloys: Alloy design

Nickel- as solvent in high temperature alloy: Justify the role of Ni

Nickel

- FCC tough, ductile
- larger cohesive energy/ outer $3d^9$ electron
- low rate of diffusion/ thermally activated creep
- no phase transformation upto T_m

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Source Book: Reed, The Superall
Fundamentals and Applications, C

So, now we understood that why nickel is chosen for any solvent atom in a nickel super alloys. Because it is FCC structure it is tough and ductile throughout the temperature range of operation. Now it has a larger cohesive energy because of the outer 3-d 8 or 9 electrons. And it has a very low rate of diffusion and thermally activated creep because creep is one of the most important properties to be consider as a material for high temperature. And the most important thing is that that up to the melting temperature there is no phase transformation in nickel. And this is very important aspect that there is no degradation of the properties or alteration of the properties as far as nickel is concerned.

Now, let us try to look at what are the other alloying element should be considered for strengthening nickel alloys.

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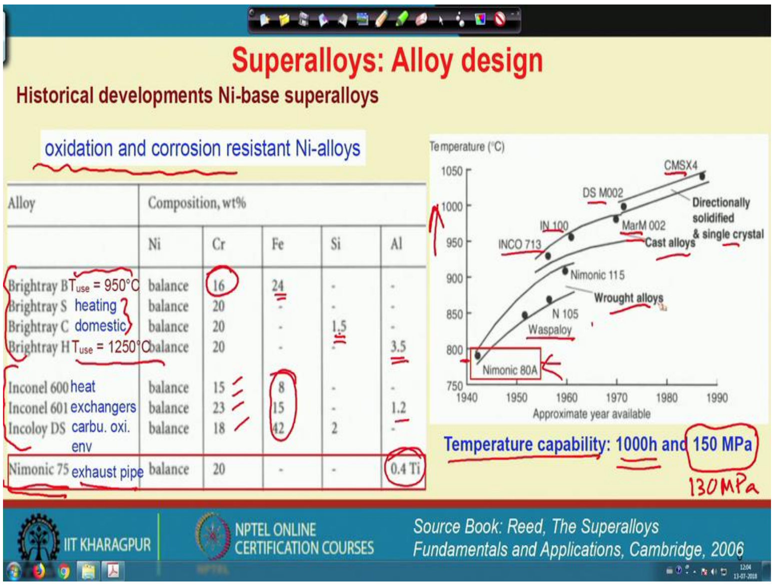

Superalloys: Alloy design

Historical developments Ni-base superalloys

oxidation and corrosion resistant Ni-alloys

Alloy	Composition, wt%				
	Ni	Cr	Fe	Si	Al
Brightray B T_{use} = 950°C	balance	16	24	-	-
Brightray S heating	balance	20	-	-	-
Brightray C domestic	balance	20	-	1.5	-
Brightray H T_{use} = 1250°C	balance	20	-	-	3.5
Inconel 600 heat	balance	15	8	-	-
Inconel 601 exchangers	balance	23	15	-	1.2
Incoloy DS carbu. oxi. env	balance	18	42	2	-
Nimonic 75 exhaust pipe	balance	20	-	-	0.4 Ti

Temperature capability: 1000h and 150 MPa

130MPa

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Source Book: Reed, The Superalloys Fundamentals and Applications, Cambridge, 2006

And for that we have to look into the history of the superalloys. To see how these alloys have been developed with time; so even around 1940s or so, or earlier time. This alloy was called bright alloys are developed.

So, here we are only talking about oxidation and corrosion resistance of nickel base alloy. I am not talking about in terms of their creep properties actually, as stress is there. So, here this bright alloy the maximum working temperature was something like 950 °C, where chromium and iron were added. And these are used mostly for heating purposes for some domestic appliances or industrial appliances. Silicon was little bit used and let us say aluminium also used later on for a little bit higher application temperature; where chromia scale does not perform better.

Now I am with the time for the heat exchangers and let us say a carburizing atmosphere or let us say oxidation environment, in both cases the same alloys this Inconel alloys has been developed. The Inconel also content here chromium let us say in between 15 % to 20 %.

So, iron is also added and also some aluminium. So, along this direction, the Nimonic of one of the series that came up; which use for let us say exhaust pipe. Where also we need some good high temperature properties, where some amount of titanium is also added. And if we consider in terms of high temperature capability by considering creep. So, the benchmark was in between let us say 1000 hours of application, and at a stress of 130 to 150 mega pascal.

So, in that particular range, you see even though Nimonic we considered as to be used let us says above 1000, but when stress is applied the usable temperature range here is somewhat 800 °C. And Waspaloy we have already talked about where we have use the wrought alloys. And Waspaloy was very famous for these wrought alloys or let us say directly cast alloy like here INCO 713.

And these alloys or let us say some of these MarM alloys or let us say directionally solidified alloys or CMXS series. However, these alloy has been developed later on to improve their operating temperature in presence of stress by only the development of some processing condition considering some of the alloy addition. Means, processing condition has been improved, compared to wrought alloy, in cast alloy we get equiaxed grain.

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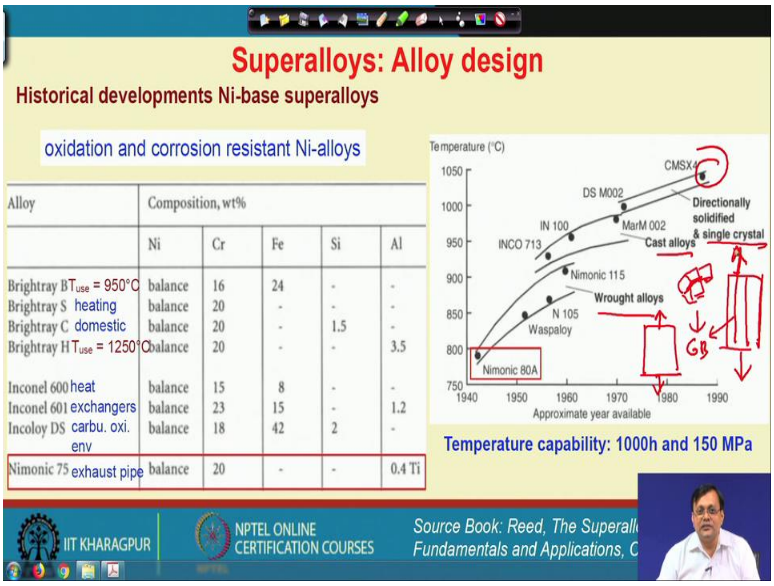

Superalloys: Alloy design

Historical developments Ni-base superalloys

oxidation and corrosion resistant Ni-alloys

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Brightray C domestic	balance	20	-	1.5	-
Brightray H T_{use} = 1250°C	balance	20	-	-	3.5
Inconel 600 heat	balance	15	8	-	-
Inconel 601 exchangers	balance	23	15	-	1.2
Incoloy DS carbu. oxi. env	balance	18	42	2	-
Nimonic 75 exhaust pipe	balance	20	-	-	0.4 Ti

Temperature capability: 1000h and 150 MPa

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Source Book: Reed, The Superall
Fundamentals and Applications, C

So, equiax grain means whenever we will apply stress, here will be the break. However, if we take some directionally solidified grains. If we apply, stress in this direction the grain boundary along this way normal to this direction are very less.

So, these alloy such and microstructure will survive. So, I am talking about the grain boundaries. And if we simply make a single crystal, where there is no grain boundary at all, then definitely they will perform much better and this CMXS series alloys are used for the blade application. But besides blade application, we can use these alloys. So, blade is a very precise thing, but cast alloys can be used for other application also. So, that is why this development basically come along from these bright alloy towards this from these 1940's up to today, and now there are several elements.

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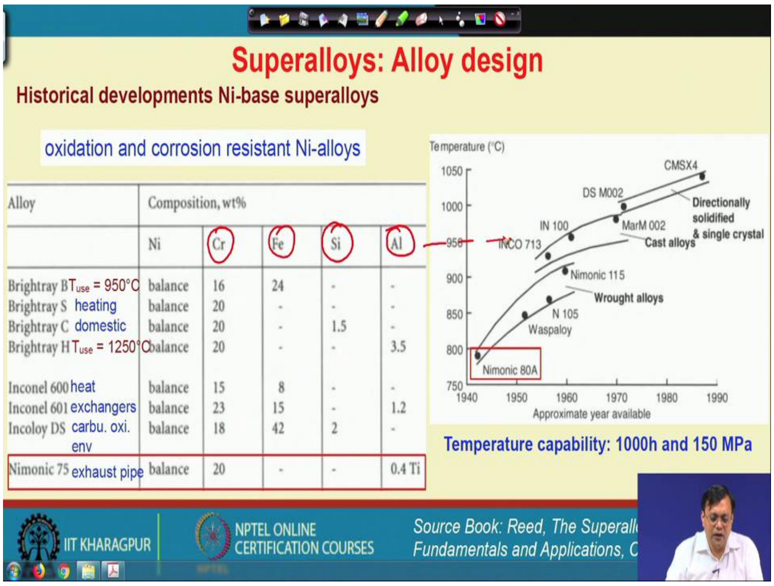

Superalloys: Alloy design

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Temperature capability: 1000h and 150 MPa

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Source Book: Reed, The Superall
Fundamentals and Applications, C

You can always ask what is the role of these elements, or maybe some other alloying element that has been added. So, I if you consider that then there is very specific purpose of these alloy addition.

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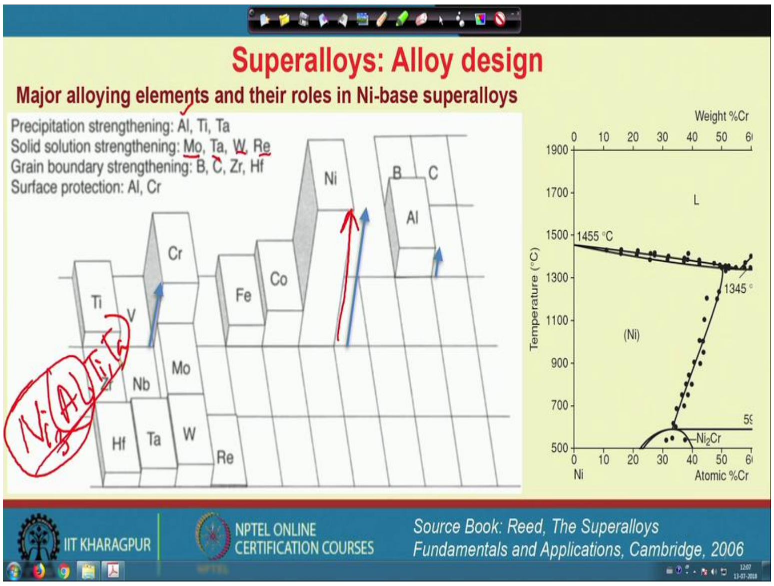

Superalloys: Alloy design

Major alloying elements and their roles in Ni-base superalloys

Precipitation strengthening: Al, Ti, Ta
Solid solution strengthening: Mo, Ta, W, Re
Grain boundary strengthening: B, C, Zr, Hf
Surface protection: Al, Cr

$Ni_3(Al,Ti,Ta)$

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Source Book: Reed, The Superalloys
Fundamentals and Applications, Cambridge, 2006

Where nickel is the majority of the concentration; however, for solid solution strengthening which is one of the major strengthening issues, where molybdenum, tantalum. And later on these tungsten and rhenium has been discovered to be added in this particular purpose.

So, I have a solid solution strengthening. And one of the most important precipitation hardening is by the aluminides in the nickel base alloy. So, I have Ni_3Al precipitate which is required for strengthening purpose. So, there we use aluminium purposely so that we form this particular precipitate as strengthening for high temperature. And also sometimes titanium is added there or tantalum.

So, we preferred these kinds of complex or other alloying elements which goes inside the precipitate. Now the third purpose of other alloying elements likes some amount of boron, carbon which are added in very little amount like 0.2 *wt. %* or less so to form carbide and strengthen the grain boundaries. And now there is only 2 elements available for surface protection or oxidation like aluminium and chromium. For much higher temperature aluminium is mostly preferred.

So now you understood that in nickel where majority of the alloying element chromium is added, for a purpose that it will strengthen the alloy in terms of their surface protection. And we can go for let us say up to 20 %. So, this will provide a production of 1000 °C, very good because of the chromia formation. So, this is a purpose of chromium in nickel superalloy and these are the directions shown in terms of the percentage of the element that has been added. And now let us consider what are the other role and continue this.

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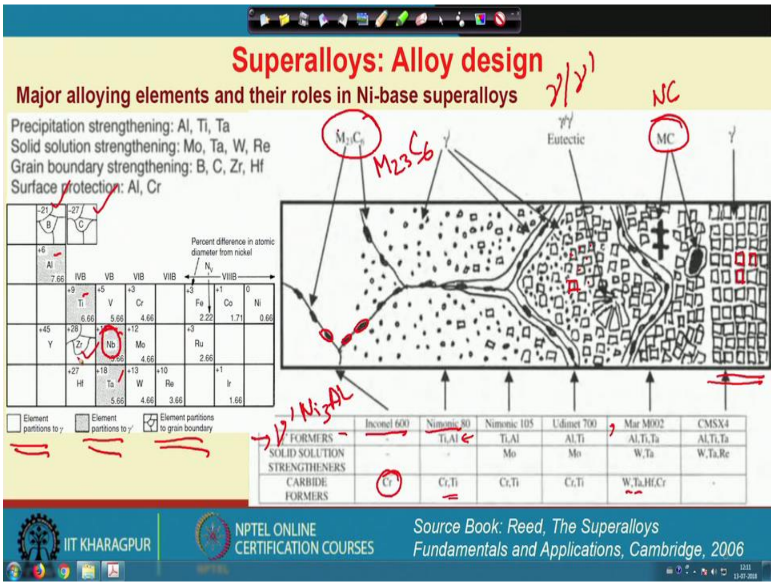

Superalloys: Alloy design

Major alloying elements and their roles in Ni-base superalloys

Precipitation strengthening: Al, Ti, Ta
Solid solution strengthening: Mo, Ta, W, Re
Grain boundary strengthening: B, C, Zr, Hf
Surface protection: Al, Cr

	Inconel 600	Nimonic 80	Nimonic 105	Udimet 700	Mar M002	CMSX4
γ' FORMERS	-	Ti,Al	Ti,Al	Al,Ti	Al,Ti,Ta	Al,Ti,Ta
SOLID SOLUTION STRENGTHENERS	-	-	Mo	Mo	W,Ta	W,Ta,Re
CARBIDE FORMERS	Cr	Cr,Ti	Cr,Ti	Cr,Ti	W,Ta,Hf,Cr	-

Source Book: Reed, The Superalloys Fundamentals and Applications, Cambridge, 2006

The wide range of microstructure that has been prepared in these nickel base superalloys: so we have the γ′ former. γ′ is basically the phase that is Ni_3Al. This is an ordered intermetallic phase inn Inconel 600 or Nimonic series. In Inconel we do not add this, whereas the microstructure mostly contains these kind of precipitate at the grain boundaries; which are $M_{23}C_6$, M 3C6. And these are carbide, and these carbides form during hardening or any treatment or during service itself and strengthen the alloy.

Where the carbide former chromium is required which is replacing the m here actually. Now for Nimonic we a purposely at titanium, aluminium to form this γ′ phase and also carbide formers are used whereas, in case of Nimonic 105 or udimet. We have such kind of eutectic like microstructure containing γ and γ′ phase.

So, these are the γ′ phase. In a matrix, that is the γ-nickel. Now I am also in this Mar group of material where there are some metal carbides MC that form actually where tungsten and tantalum are used. Tungsten carbide is here you know a chemistry of this tungsten carbide. And for a CMXS, these are the very advanced series, which are used for blade, where such kind of unique microstructure of γ′ phase embedded in a γ-nickel matrix is preferred.

So, we understood that at these particular elements where the element need to be partition in γ, or element need to be partition in γ′ or element used for the grain boundary like boron, carbon or zirconium for the grain boundary precipitate strengthening purpose. Or let us say these are the element which mostly contribute to partition in the γ′ or goes into γ′. niobium usually does not use in nickel, but in iron-nickel superalloy, niobium is used where the element goes into the γ phase or are these elements like vanadium, chromium. So, we already understood the role of addition of various alloying element in this superalloys.

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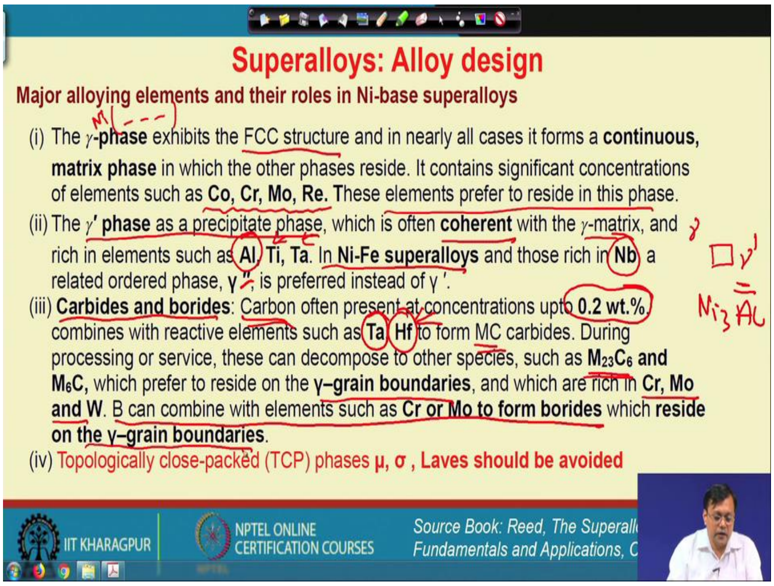

Superalloys: Alloy design

Major alloying elements and their roles in Ni-base superalloys

(i) The γ**-phase** exhibits the FCC structure and in nearly all cases it forms a **continuous, matrix phase** in which the other phases reside. It contains significant concentrations of elements such as **Co, Cr, Mo, Re.** These elements prefer to reside in this phase.

(ii) The γ' **phase** as a precipitate phase, which is often **coherent** with the γ-matrix, and rich in elements such as **Al, Ti, Ta**. In **Ni-Fe superalloys** and those rich in **Nb** a related ordered phase, γ '', is preferred instead of γ '.

(iii) **Carbides and borides**: Carbon often present at concentrations upto **0.2 wt.%** combines with reactive elements such as **Ta**, **Hf** to form MC carbides. During processing or service, these can decompose to other species, such as $M_{23}C_6$ **and** M_6C, which prefer to reside on the **γ–grain boundaries**, and which are rich in **Cr, Mo and W**. B can combine with elements such as **Cr or Mo to form borides** which **reside on the γ–grain boundaries**.

(iv) Topologically close-packed (TCP) phases **μ, σ , Laves should be avoided**

Source Book: Reed, The Superall
Fundamentals and Applications, C

So, if I like to summarize today whatever we have discussed. So, we need a γ matrix which is FCC structure and this is a γ-nickel phase actually. And where several other alloying elements strengthens by solid solution strengthening. For solid solution strengthening, we add basically cobalt, chromium, molybdenum, or rhenium. And these elements prefer to reside inside that γ phase, whereas inside a γ matrix we add a γ′ phase. We prefer that this γ′ phase should form. And this is a precipitate phase which is often coherent. And you understand the coherent precipitate require very high strength and strengthening effect is very good inside the γ.

So, this is a ordered FCC phase. And these phase particularly since it is Ni_3Al phase, and rich in aluminium means 25 % aluminium is here; however, we also add some amount of titanium and tantalum to further strengthen. So, in case of nickel-iron superalloy, here niobium is often used which is related with the formation of γ′′ phase.

Now for grain boundary strengthening, for at higher temperature the carbides and borides are preferred. So, here to form carbide, tantalum and hafnium are used for MC type of carbide, or let us say 0.2 % carbon is added which will form the carbide along with this element. However, $M_{23}C_6$ type of carbide.

So, these are also form at the grain boundaries of γ phase. Chromium, molybdenum and these elements are used. However, sometimes boron is also used which will form boride with the chromium and molybdenum and reside in the γ-nickel grain boundaries. However, whatever alloying elements we have added for a very specific purpose, we must keep it in mind that there should not be any topologically close pack phases, because it enhances embrittlement.

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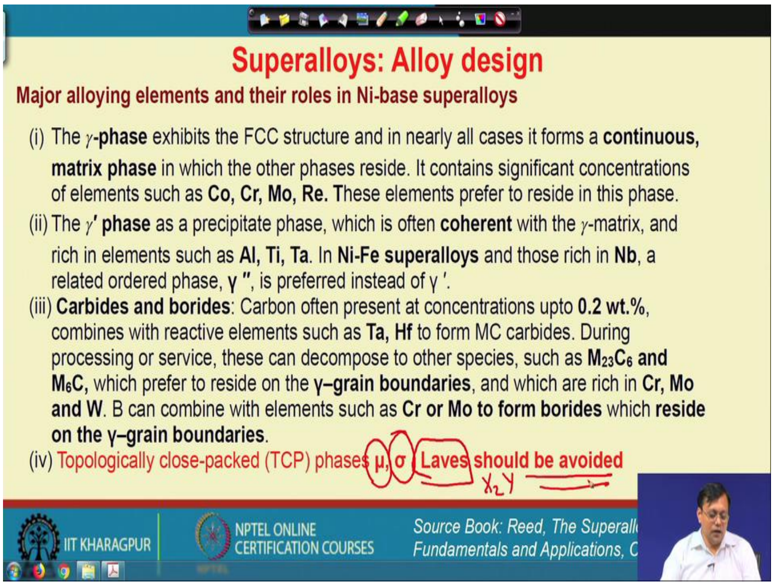

Superalloys: Alloy design

Major alloying elements and their roles in Ni-base superalloys

(i) The γ-**phase** exhibits the FCC structure and in nearly all cases it forms a **continuous, matrix phase** in which the other phases reside. It contains significant concentrations of elements such as **Co, Cr, Mo, Re.** These elements prefer to reside in this phase.

(ii) The γ' **phase** as a precipitate phase, which is often **coherent** with the γ-matrix, and rich in elements such as **Al, Ti, Ta**. In **Ni-Fe superalloys** and those rich in **Nb**, a related ordered phase, γ'', is preferred instead of γ'.

(iii) **Carbides and borides**: Carbon often present at concentrations upto **0.2 wt.%**, combines with reactive elements such as **Ta, Hf** to form MC carbides. During processing or service, these can decompose to other species, such as **$M_{23}C_6$ and M_6C,** which prefer to reside on the **γ–grain boundaries**, and which are rich in **Cr, Mo and W**. B can combine with elements such as **Cr or Mo to form borides** which **reside on the γ–grain boundaries**.

(iv) Topologically close-packed (TCP) phases μ, σ, **Laves should be avoided**

X_2Y

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And we have new phase and sigma phase all Laves phase. And these Laves phase means basically X_2Y type of phase and A_2B type of compound that should be avoided. So, this is all about the superalloys in case of a nickel base super alloys and the alloying elements addition. And we will go to detail of the physical metallurgy in terms of microstructure mechanical properties or high temperature properties specifically in the next class.

Thank you very much.