

Advanced Materials and Processes
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Lecture – 27
Introduction to High Temperature Materials (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. Last classes, we have just started our discussion on introduction to high temperature materials. Today, we will continue the discussion. In the last class, we have talked about what are the main and important characteristic a high temperature materials should exhibit, or engineers before design an application of a material at higher temperature.

What are the characteristic they really look for? However, material may satisfy our criteria or may not, but if a material can satisfy our criteria, can we increase the capability of that material to a much better condition? That is one of the question, whether we can use our engineering, and science knowledge, and bring changes into the material so that they could serve our purpose in a better way, or a better service life could be expected from a material.

In that case we have to learn some of the very important aspects of metallurgy. Today, we will discuss those things, means how to enhance the capability of a high temperature material for a better service.

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High Temperature Materials: Enhanced Capability

- Metallic Materials/Alloys**
 - solid solution strengthening
 - precipitation strengthening
 - dispersion strengthening
 - grain size and GB effects
 - environmental resistance
- Ceramic Materials**
 - phase control
 - defect tolerance
 - thermal shock resistance
- Composite Materials**
 - Titanium matrix (requirements: strength, stiffness)
 - Carbon-carbon (requirements: defect tolerance, reliability, stiffness, oxidation)
 - Ceramic matrix (requirements: defect tolerance, reliability)

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Since we mostly talked about metals and alloys, there are some of the mechanisms that play a very crucial role on their capability to use at a high temperature. So, here I have just listed those particular mechanism that plays important role in enhancing the strength and for their other mechanical aspects, like solid solution strengthening. Solid solution strengthening means we add a solute atom in order to increase the lattice friction as well as it simply stop some of this dislocation, the friction stresses also increases.

Now, in case of precipitation hardening, the precipitate act as an obstacle for dislocation movement and increases the strength. Dispersion hardening where precipitate gets dissolved at higher temperature, dispersion strengthening is done using some second phase oxide or nitrite or some ceramic particle in a metal matrix.

Now, there could be effect from the grain size and grain boundary and environmental resistance. So, in other class of material, there are some other important aspect like ceramic material which is also used for high temperature application, where the volume fraction of different phases, where defect tolerance has to be considered or let us say thermal shock resistance.

Now, in case of composite material, like I have a metal matrix and we incorporate some other second phase ceramic fibre or particle in order to exploit those material for a higher temperature. And there, we have a small window of enhancing the capabilities in terms of their strength, by controlling the volume fraction of the second phase. Or, let us say the

stiffness whether we can enhance those property or not. And that is why I write here as a enhance capability, means it has already some capability. However, we want to enhance the capability for a better service life.

Now, in case of carbon-carbon composite, usually people or engineers look for a better defect tolerance, and to get a reliability of the processing and application; whether you can use the stiffness. And since the material contain carbon, once it is exposed to a relatively higher temperature carbon form carbon dioxide, carbon dioxide is volatile in nature. So, immediately there is a degradation occur, you understand the matter now.

So, the oxidation aggrieves, now in case of ceramic matrix, again the same question comes, the defect tolerance. So, this is a very common aspect for any kind of ceramic material for defect tolerance, why? Because a ceramic has a lower fracture toughness, I discussed in a very initial days like K_{IC} . So, here, if the fracture toughness is very low and if it contain very small crack that can directly cause to a failure, the critical crack length size is very small, in the range of a few micrometre. And also the stress redistribution also decreases.

So, we need some reliability. So, we will discuss these aspects today in detail.

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High Temperature Materials: Enhanced Capability

Metallic Alloys: solid solution strengthening

- internal strains in the parent lattice due to solute atoms result in strengthening
- atoms with similar crystal structure and lattice parameter exhibits high mutual solubility and will generate relatively little strengthening. Atoms of different size may have limited solubility but could generate significant strengthening
- W, Mo in Ni-superalloys
- Re decreases the diffusion and retard coarsening of γ' in γ -Ni matrix of superalloys

Change in 0.2% PS (MPa)

Alloying element (at %)

Effect of alloying element on PS of γ -iron

Source Book: Meetham et al, Mat Temperature Applications, Spring

Let us first start with the metallic alloys in terms of solid solution strengthening. And if we add a solute atom, it causes strengthening. There is no doubt this is from a very basic understanding of metallurgy, physical metallurgy that we have learnt.

However, the internal strain here, plays the crucial role in the parent lattice due to the presence of the solute atom that causes the strengthening. However, the atoms with similar crystal structure, and very close lattice parameter exhibit higher mutual solubility, like I can simply think about copper and nickel. However, they will generate a relatively small strengthening effect, because they have the same crystal structure and the lattice parameter difference is less. And that will cause a lesser internal strain in the material.

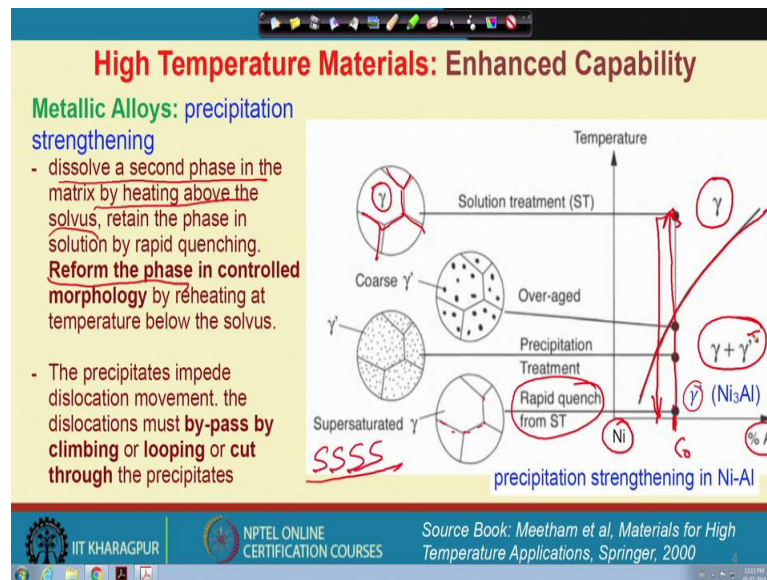
So, an atoms with larger difference in their size will have a limited solubility, but it could generate a significant strengthening effect. So, one has to choose which solute atom we really incorporate up to what extent, means I am talking about concentration to generate how much increment in the strength. So, let us talk about the change of the strength. So, this is not the absolute value of the proof stress or yield strength in a γ -iron. However, we are talking about the $\Delta\sigma$.

So, this is the percentage or atomic percentage of alloying element. If we add vanadium then there will be slight rise. However, if we add nitrogen, you see it has a very sharp rise. So, this is just an example that I like to show you that how the solute atom can changes the yield strength depending on the concentration and their relative size difference.

Now, in case of nickel based super alloy it is very common to add tungsten and molybdenum. So, tungsten and molybdenum are added purposely in order to enhance the strengthening by solid solution strengthening. However, these days rhenium is also added which has some different effect not only solid solution strengthening, but also it decreases the diffusivity and also it retard the coarsening effect. So, if you have precipitate, if you have grains, they may coarsen. So, in that case if we can simply stop this coarsening then we can exploit the coarsening effect of let us say the precipitate which is γ' -Ni₃Al phase in a γ -nickel matrix in the nickel based superalloys.

So, you can see that the very basic engineering comes from the alloy addition, and how the alloy addition can changes the relative strength in a lattice.

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Now the second issue is the precipitation strengthening. So, by precipitation strengthening, we can enhance the strength of an alloy. How we do that? It is very common age old strategy, like we dissolve a second phase in a matrix by heating above the solvus. How? We should see the phase diagram. And here I show you a nickel and aluminium phase diagram. So, this is the solvus line. So, here is the γ phase is stable and here is the γ and γ' is stable.

Let us assume that I have chosen a composition here it is C_0 , and now I simply raise the temperature to a γ -phase field. In that case, I will have grains with γ only; this is the grain boundaries of γ . Now, this is called solution treatment; means, I reached to a single solute solution phase where we are holding it without allowing too much grain growth, and in order to homogenize and dissolve the precipitate which appeared initially.

After that we simply quench it to a room temperature. And after quenching what we get by this rapid quenching from the solution treatment, we get a supersaturated solid solution. This is called supersaturated solid solution. Here also we get γ . However, all aluminium is inside this γ -solid solution, which is higher than the equilibrium condition.

Now, what we do? We basically reform the phase means; the γ' phase should again appear by heating at a temperature below the solvus. So, we take again this C_0 , and we simply warm it up to different temperature for different time for precipitate to appear in the microstructure. And if we heat it at a relatively higher temperature very close to the solvus,

then we will get a larger size γ' phase. Whereas if we heat it to a relatively lower temperature, then we will get finer precipitate, and finer more number.

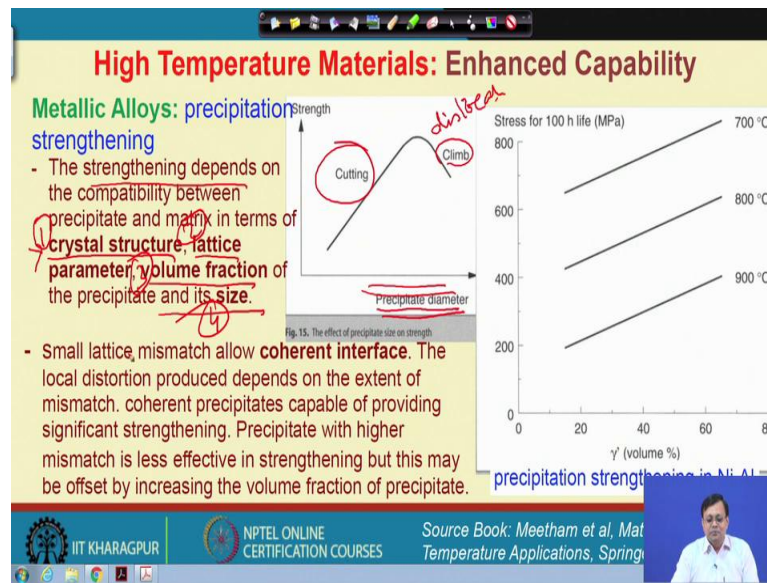
And if we have a lower than the, nucleation will be higher and higher, and the diffusivity will be less. So, there particles will be close to each other, but at higher temperature since diffusion length is higher and higher. So, we will get a relatively less number of nuclei and a coarser microstructure, means coarser precipitate.

Now, this is we call it somewhat like a overaged or this is let say what we really desired as a precipitation treatment that is necessary, why because, the size of this precipitate has some role on the mechanism, because it control the mechanism of strengthening, how? This precipitate actually impede the dislocation movement. The main reason of this precipitate when the dislocation will appear it will create as an obstacle.

So, for that particular purpose the dislocation may bypass by climbing or it may leave a loop around the particles, or it can cut through. How does it occur? I have a particle here I have the dislocation, which is trying to pass and this is the precipitate, the dislocation may leave a loop and then dislocation comes here. Or it can simply cut through the precipitate. So, this is let us say the precipitate and then after cutting it will be something like this and here is the dislocation so, this is like the situation.

So, we can think about which one is better in terms of controlling the mechanism. So, we will see that how to alter and how to control such a circumstance and situation.

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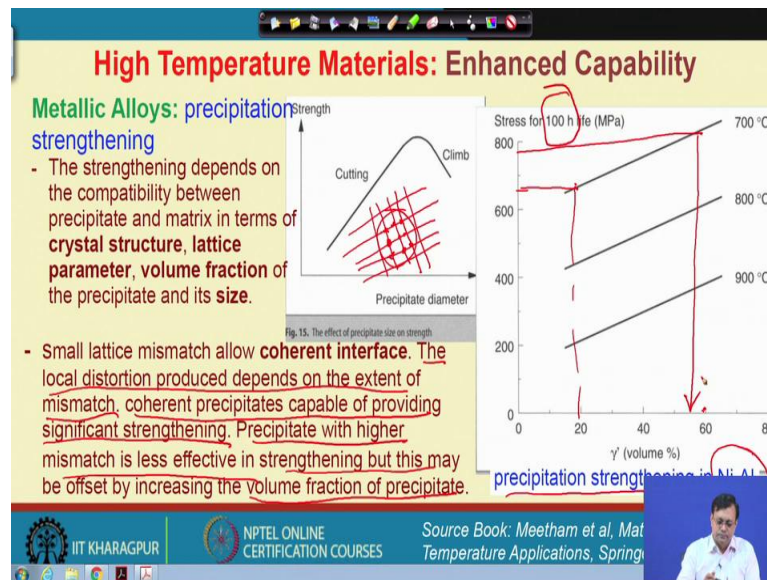
So, in case of precipitation strengthening, we have seen that if the precipitate diameter is very large, then it basically bypass or it will climb the dislocation, this is a dislocation climb action. Or dislocation will cut through the precipitate when the precipitate is smaller in size.

So, this strengthening actually depends on the compatibility between the precipitate, and the matrix in terms of their crystal structure, because the parent crystal structure may be different than the precipitate crystal structure. Or they may be both are let us say FCC phase; however, the lattice parameter may be changed because if lattice parameter change then the coefficient of coherency of the precipitate or non-coherency or incoherent or semi coherent type of precipitate may appear.

And also there are volume fraction of the precipitate that control the size of the precipitate. Here, I just show you in terms of their size. However, one has to consider all these aspects in terms of crystal structure, lattice parameter, volume fraction, and the size. All this parameter will control the mechanism that govern the strengthening effect.

So, as an example, I can tell you like a small lattice mismatch. We will create a coherent precipitate what does it mean?

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That I may have a lattice, and this is the lattice point, and here this is the solute atom, which make a cluster to make a precipitate. So, this is almost like a coherent precipitate, now in such a case the local distortion depends on the extent of the mismatch. There is no doubt because the lattice mismatch will create the lattice distortion.

Now, coherent participate are capable of providing a significant strengthening effect. But precipitate with higher mismatch is less effective in strengthening, because then the dislocation will bypass and the strengthening can only be achieved by increasing the number of the precipitate, or let us say the volume fraction of the precipitate, when the precipitate has a higher mismatch. So, this is let us say an example of such kind of precipitation strengthening that we talked about.

So, I show you another example in case of nickel aluminium, that if we need a nickel aluminium alloy to use for a higher temperature, at relatively higher stress let us say we fix here for a 100 hours of life. Then for a lower precipitation volume fraction we will get a lower strength. However, if we need to use for a higher temperature, then we have to go for a higher precipitate volume fraction.


So, here is the situation, where precipitate with higher mismatch will be less effective and we can control that using the volume fraction of the precipitate. Now if we have to go for a higher temperature then again for higher stress application we have to go for higher fraction of the precipitate. So, this is the control that one should look for.

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High Temperature Materials: Enhanced Capability

Metallic Alloys: dispersion strengthening

- The precipitates progressively coarsen and re-dissolve at higher temperatures.
- Fine, uniform distribution of dispersoid particles that are essentially insoluble in the matrix.
- Al_2O_3 in Al-matrix or TD-Nickel: ThO_2 in nickel
- The dispersoid phase is not coherent with the matrix and so the strengthening effect is not as pronounced as with precipitation hardening but it is maintained to higher temperatures.



grain size and grain boundary effects

- GB is weak at $>0.5T_m$ and strong $< 0.5T_m$
- Hall-Petch at low T
- at high T, carbide/boride pinning at GB hinders GB sliding, better creep resistance

	Conventional forge + heat treat 1090 + 775+705 °C	Modified Forge +HT980+720+650 °C
Grain Size	ASTM 2	ASTM 6
0.2% FS (MPa) 575 °C	752	839
LCF (0-850 MPa) 500 °C	$>10^8$ cycles	$>3 \times 10^4$ cycles

	UDIMET 500 Base Alloy	UDIMET 500 - Base Alloy +0.19%Zr +0.009% B
Creep life (h) 175 MPa 870 °C	50	650
Rupture ductility (%)	2	14

Source Book: Meetham et al, *Mat Temperature Applications*, Springer

Now, let us have a look at how to enhance the capability using some dispersion strengthening. So far we have talked about solid solution strengthening, and the precipitation strengthening here, if we have a precipitation strengthen alloy then we can raise the temperature to a single phase we can dissolve it. Or this is purposely dissolve in order to tune the microstructure.

But it may also occur that I am using an age hardenable alloy, this is called as precipitation strengthen or age hardenable alloy at a higher temperature. During its service the precipitate may grow, in that case, the strengthening mechanism will change and the service life will degrade or will lower down, because the precipitate may also dissolve in the matrix at higher operating temperature.

So, in order to prevent that, one of the alternative solution is to go for dispersion strengthening. So, here this dispersion strengthening is a particular terminology we use where we incorporate a second phase which is very small, but they are non-metallic, non-metallic in bonding, means they are oxide, nitride, and ceramic particle. So, this precipitate they usually do not grow, and the progressive coarsening will never be occurred in case of ceramic. So, here we employ a fine and uniform distribution of dispersoid particle that are essentially insoluble in the matrix.

So, there are very nice example like we know aluminium-alumina composite. So, I have an aluminium matrix, inside I have precipitate, which are Al_2O_3 in an aluminium matrix.

So, aluminium matrix has some grains, and I may have inside this grain there are some alumina precipitate.

So, alumina precipitate is ceramic in nature, and they will not grow at all, the growth is very less. And they are also not soluble. Under such circumstance, we call it as a dispersion strengthening alloy. Another very nice example is the thoria dispersed nickel, TD nickel, the TD nickel has thoria particle in a nickel matrix. So, this dispersoid phase is not at all coherent.

Because they are ceramic and they have a very different crystal structure, lattice parameter than the matrix aluminium or nickel matrix. And it is not pronounced as precipitation hardening. In case of precipitation hardening, we can increase the strength to a very high extent, but it is maintained, the interesting point here that we can allow this composite to be used at higher temperature; because the precipitates are very much or dispersoids are very much stable.

Now, the other way of enhancing the capability is by controlling the grain size and grain boundary effect. Because once we have talked about the second phase, because in terms of dispersion strengthening and solid solution strengthening, and let us say the precipitation hardening.

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High Temperature Materials: Enhanced Capability

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Creep life (h) 175 MPa 870 °C	50	650
Rupture ductility (%)	2	14

Source Book: Meetham et al, Materials for High Temperature Applications, Springer, 2000

However, in a base alloy there are grains, right? And once these grains are exposed to higher temperature the grain growth will occur because the grain boundary will migrate, that is that will occur yes by diffusion.

So, this grain boundary has a positive effect at lower temperature, because the grain boundary is strong below the homologous temperature, or equi-cohesive temperature. Equi-cohesive temperature means where the grain body and grain boundary, they have the same strength level. So, this is called as equi-cohesive temperature. So, this is usually at a $0.5 T_m$ is the melting temperature multiplied by 0.5.

Now, the grain boundary are usually weak at a temperature above $0.5 T_m$, and very strong at a $0.5 T_m$. It simply said that a finer microstructure will have a better strengthening effect at lower temperature, and a coarser microstructure has a positive effect as a high temperature material at higher temperature. And the reason is very clear, because we all know about Hall-Petch equation. So, in a Hall-Petch equation: $\sigma = \sigma_0 + \frac{k}{\sqrt{d}}$ where σ is basically is a proportional where there is a constant into inverse of \sqrt{d} .

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High Temperature Materials: Enhanced Capability

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Source Book: Meetham et al, Mat
Temperature Applications, Spring

So, here this is let us say a friction stress, and dependent on the grain size; where k is the hall-Petch constant, right?

So, in that case, if we can increase the population of the grain boundary or finer grain size, then ASTM grain size, this is as an example that we can increase the strength much and

much higher. ASTM grain size is larger grains. This is let us say ASTM 2 and the very finer grain is the ASTM 8 finer grain or ASTM 6.

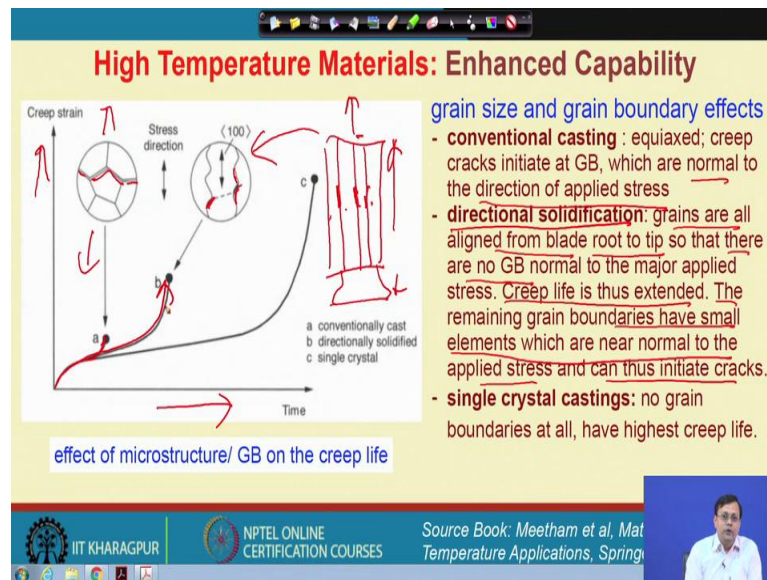
So, here we see that the proof stress at 575 °C, it is 752 where as a finer grain size has a higher proof stress at this given temperature, which is good. However, you can see the major benefit, that you can simply increase the fatigue life from 10^4 cycle to 3×10^3 times higher a fatigue cycle can be achieved a finer grain size.

Now, at higher temperature the grain boundary is the weaker region. So, what we can do? We can simply take this material, and intentionally put some precipitate at the grain boundary. What it does? It simply impede the motion of the grain boundary, it will be stopped. Because they are pinning the grain boundary, if grain boundary has to move they have to move along with that. That will be a good purpose. How to do that? We can incorporate some carbide or boride particles by grain boundary engineering, and incorporate those precipitate inside for a better creep resistance. As an example UDIMET is an example of such.

So, here this is the base alloy with some zirconium and boron. In that case, the creep life can be increased such a high extend you can see from 50 to 650 hour of use just by adding such kind of precipitate at the grain boundary, whereas the rupture ductility can also be improved.

So, this is also another interesting way by controlling the grain size and introducing some grain boundary effect.

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Now, how to further improve that. Now the second part I talked about that was a creep. Because creep is also important phenomena for designing a high temperature alloys.

Now, here we will talk about different process in order to control the grain boundary population. How? We can cast an alloy by conventional casting method. Let us say superalloy we have cast using or any alloy, we can cast using a conventional casting, what it does? It will produce some equiaxed type of microstructure, equiaxed microstructure you know like a honeycomb structures. And this equiaxed structure means, the grain boundaries are quite large, and if we stress it, such a material then the cracks will appear at the grain boundary region.

This is normal to the direction of the applied stress, that is expected, but if we go for directional solidification, means I have a large turbine blades, and these are the grains due to directional solidification, this is a single grain, we assume. So, schematic microstructure is shown here.

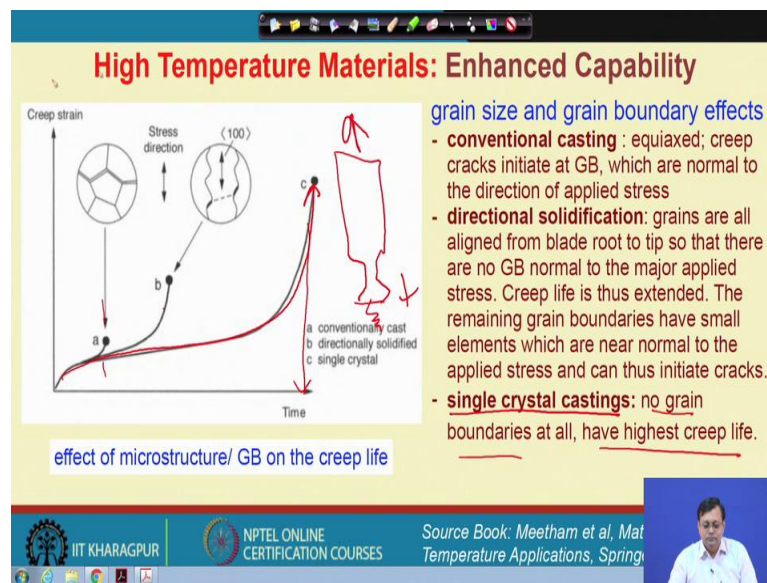
So, these are the grain individual grains, and the grains are all aligned in a blade, from the route to tip; this is let us say a route of a blade, this is from a route to the tip. And in that case there are no grain boundary that is normal to the major applied.

So, let us say the stress is applied along this way, and the creep life can be extended. Because if crack has to appear it has to appear here. So, only in such case, the crack will

appear and the remaining grain boundaries have a very small element which are near normal to the applied stress and to initiate the crack. So, you can see that this is a creep strain, and this is the creep time. So, in case of conventionally cast alloy, the performance is poor than the directionally solidified alloys.

So, here we get a better service life; however, on the other case we can go for a single crystal casting so or growth. So, in that case there are no grain boundary at all.

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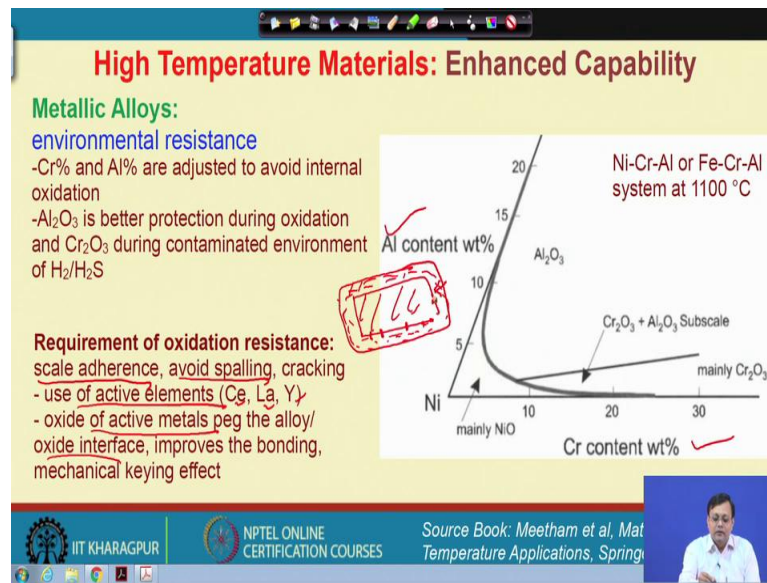


So, the whole turbine blade is made out of a single crystal only. This is a single crystal, using a seed crystal, we will going into detail of the processing of those.

So, here there is no grain boundary at all. So, if we apply some stress, then crack will be very difficult and there will be no grain boundary. So, these is no grain boundary diffusion, only grain body diffusion. That can be controlled by adding some rhenium. So, this will be a very best material for a highest creep life.

You can also see schematically I have shown you here that you have a very large time compared to any directionally solidified or let us say conventionally cast alloys.

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Now, there are some other aspects like environmental resistance, which we partly talked about; however, for enhancing the capability we can choose the alloying element. Let us have a look at some nickel based alloy we want to add some chromium or aluminium.

People have seen that there are some optimized condition whether you want alumina on the surface after oxidation or chromium in the surface. Like in a stainless steel you already know about chromia scale which protect the surface from oxidation. So, this chromium concentration or aluminium concentration in a nickel base alloy could be adjusted to avoid some internal oxidation.

So, alumina always provide a better protection during oxidation environment and chromia during contaminated environment in presence of some sort of sulphur containing compounds. So, let us say if we increase alumina content then instead of forming nickel, oxide we will get a mostly alumina oxide on the top; whereas, if we increase chromium content we will get chromia, but there are some boundary.

So, the basic requirement is that we have an alloy and the oxide scale, this is an alloy and the oxide scale, which one will dominate depend on the aluminium content or chromium content, this is interesting part. At the same time one can think that they should be sticking to the sample surface and provide a better protection. Otherwise it does not make any sense, how to do that the scale adherence is important.

And we can adhere or we can make the adherence or improve the adherence by some sort of pinning effect. How it is that? In order to avoid spalling, we can use some active elements like cerium lanthanum or yttrium. These elements form their oxides, and this active metal take the alloy at the oxide interface by some sort of mechanical keying effect and keep the oxide layer along with the base alloy. And that is one of the very beneficial effect of using any of these active elements, like cerium and yttrium.

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High Temperature Materials: Enhanced Capability

Ceramics:
phase control
 - % of SiO₂ in multiphase ceramic controls the strength, the strength and T depends on the amount of glassy phase.

defect tolerance
 - low K_{IC}, low tolerance, sensitive to local stress concentrations
 - manufacturing methods introduce local stress leads to failure during service
 - **solution:** transformation toughening

thermal shock resistance
 ceramics can not redistribute stress during plastic deformation; therefore shock resistance is important for thermal cycling

$$\Delta T = \frac{\sigma_f(1-\nu)}{E\alpha}$$

E = elastic modulus
 σ_f = fracture stress of material
 α = coefficient of thermal expansion
 ν = Poisson's ratio

	Al ₂ O ₃	Si ₃ N ₄ hotpressed	ZrO ₂ partially stabilised	Lithium Aluminium Silicate (LAS)
Flexure strength (MPa)	350	700	200	140
α (°C ⁻¹)	7.4 x 10 ⁻⁶	2.5 x 10 ⁻⁶	4.5 x 10 ⁻⁶	-0.3 x 10 ⁻⁶
E (GPa)	380	310	200	70
ΔT (°C)	95	650	1650	4850

Source Book: Meetham et al, Materials for High Temperature Applications, Springer, 2000

Now, for a ceramic high temperature application, there are 3 important aspect that one should consider for enhancing the capability. One is the phase control, another one is the defect tolerance, and another one is the thermal shock resistance. In that case, you have seen that ceramic may contain multi-phase, and one of the phase is mostly like silica. So, this silica could be glassy or may be crystalline.

So, the strength basically depends on a certain temperature, on the volume fraction of this silica, because if silica is in the glassy forms then their strength would be different than the crystalline form. So, the amount of phases we can control for enhancing the capability for high temperature application.

Now, since the defect tolerance is very less in case of ceramic we have a very, low fracture toughness, or very less process zone size. So, during manufacturing process or shaping or any kind of other mechanical operation, the local stress distribution does not occur homogenously, which will cause stress concentration in the material, and during its use

the material fails. So, there must be solution how we do that, like transformation toughening. There is a ceramic and we incorporate some phase which will transform and absorb the shock, and absorb the stress.

Now, the other point is the thermal shock resistance. Means, if we apply heat to a ceramic material, since the conductivity of ceramic is not so high as metal, there is a temperature gradient which will cause the material to fail. In such a case, the ceramic cannot redistribute the thermal stress during also plastic deformation and shock resistance.

So, here it is the important criteria people have find out that the temperature difference that is ΔT like a shock resistance how we can estimate that a given fracture stress of a material, the Poisons ratio, E is the Young's modulus and α is a coefficient of thermal expansion will give you the how much thermal shock resistance ceramic can provide.

In such a case, I show you 3 different ceramic like, alumina silicon nitride or zirconia or let us say lithium aluminium silicate, where the ΔT for lithium aluminium silicate is the highest. So, by using some parameter we can estimate which material will provide us the better service life.

So, with this today we finish our discussion. And we will continue other aspect of high temperature material in the next class.

Thank you very much.