Advanced Materials and Processes
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Lecture – 34
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. Today we will discuss some specific application areas of nickel base superalloys for gas turbine engine and we will try to see the chronological order of the development in this specific areas not only in terms of optimization of processing condition

Also how the microstructure evolves in different processing condition, and the beneficial effect of both processing condition, optimization of the alloy composition in order to develop a specific microstructure and the volume fraction of the desired phases. In case of nickel base alloy, you know that the main hardening mechanism comes from two of the mechanisms, one is solid solution strengthening another one is precipitation hardening.

So, here precipitation is the γ' phase, that is Ni3Al. So, let us start today's discussion.

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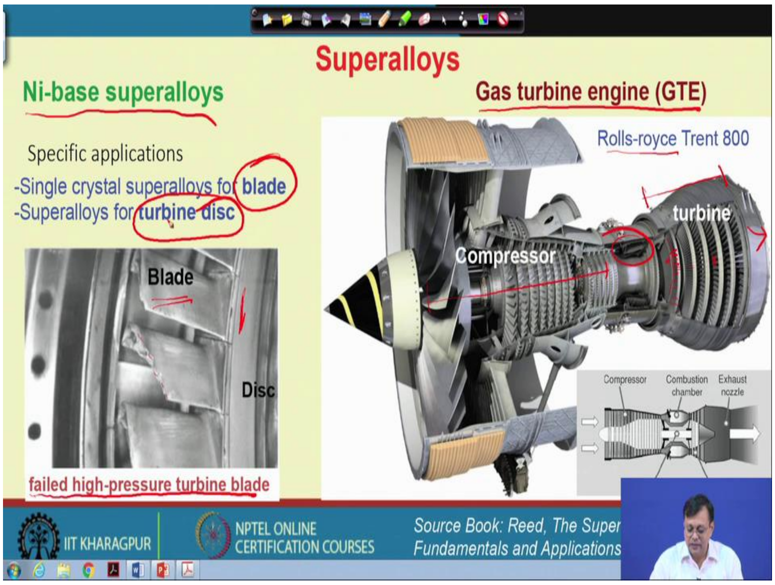

Superalloys

Ni-base superalloys

Specific applications

-Single crystal superalloys for blade

-Superalloys for turbine disc

failed high-pressure turbine blade

Gas turbine engine (GTE)

Rolls-royce Trent 800

Source Book: Reed, The Super

Fundamentals and Applications

Here in the left hand side you can see some of the failed high pressure turbine blade, and these blades are joined here with the disk. So, we have basically a disc like this and the blades are mounded on it. This is the specific design of a gas turbine engine. In the right hand side, the same sectional view of a gas turbine engine is shown. This is a specific Rolls Royce Trent 800 engine. So, the front part of the engine is the compressor and then there are combustion chamber and then the turbine.

So, here this is the turbine which is the high pressure and the low pressure or in between there are some intermediate pressure turbines and at the end it is the exhaust. Now here the front part compressor part is mostly made of titanium base alloys and here in the turbine section where the nickel base superalloys have the major use in turbine. So, here you see this is also a disk and on the disk there are blades, that are mounted.

So, in the left hand side I show you a failed high pressure turbine blade and you can see that how severe the failure has occurred. So, specifically if you look at this superalloys or nickel base superalloys, two of the major component in a turbine are made and we need a specific look into it, the second part is the turbine disk.

Now before going to the detailed discussion on the turbine disk and the blade, we first need to look at the high temperature creep behavior of the pure nickel.

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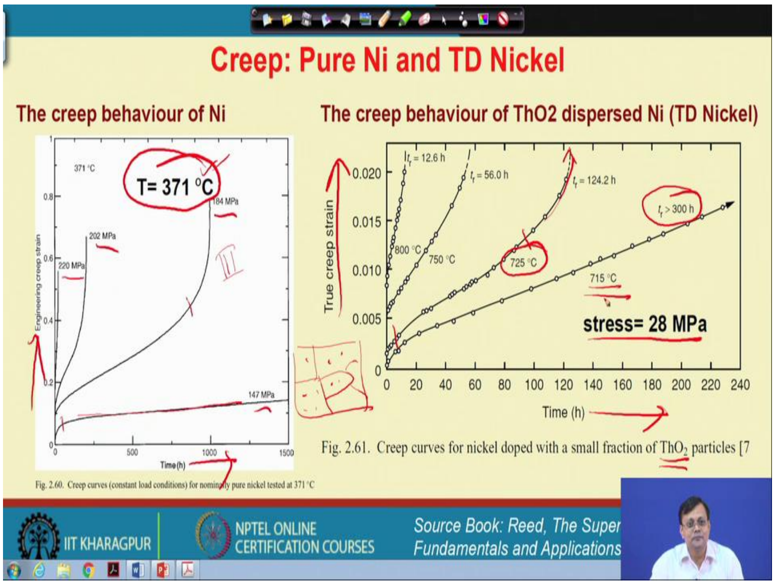

Creep: Pure Ni and TD Nickel

The creep behaviour of Ni

The creep behaviour of ThO2 dispersed Ni (TD Nickel)

Fig. 2.60. Creep curves (constant load conditions) for nominally pure nickel tested at 371 °C

Fig. 2.61. Creep curves for nickel doped with a small fraction of ThO_2 particles [7

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Source Book: Reed, The Super
Fundamentals and Applications

So, in case of nickel you will have a look, this is a plot with time versus engineering creep strain. If you look at this creep curves, here the primary, secondary and the tertiary all parts are available at different stress level. So, this is the creep test has been done as it a fix temperature of 371 °C, and the stress has been varied to develop this creep curves.

Now, the steady state you can achieve at 147 MPa at this temperature. Now if we add a ceramic particle and we have already discussed that TD nickel Thorium dispersed nickel means I have a nickel matrix and I incorporate some thoria particles in it. So, TD nickel is a very good creep resistance alloy. So, in the right hand side, the stress is fixed however, we try to measure the maximum time to rupture means until what time the material survives and then it fails.

So, the rupture time in case of a higher temperature is something like 725 °C, you can have a look that it basically gives some failure. So, this is the total creep strain and here in the X-axis this is the time. So, in case of Thoria disperse particle you can go up to higher elevated temperature and one must look at what are the mechanism involved in such a high creep resistance because in nickel base superalloy we purposely chosen nickel which is a fcc crystal structure as a matrix.

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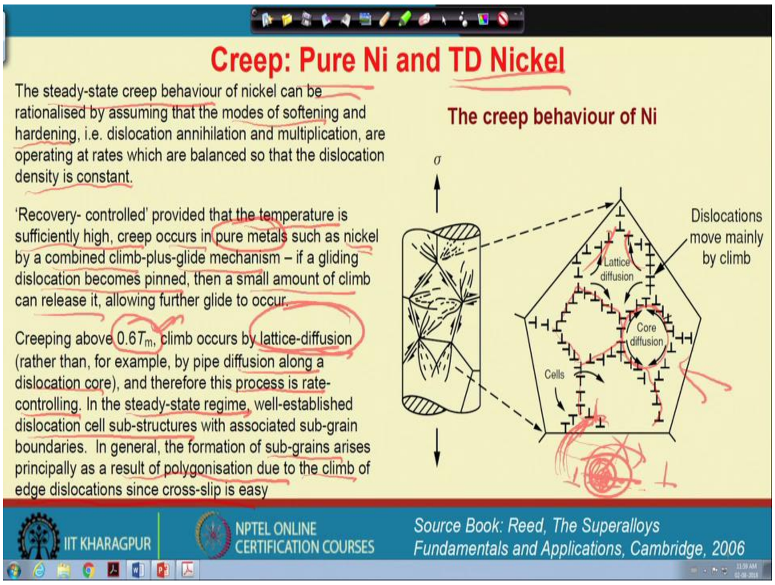

Creep: Pure Ni and TD Nickel

The steady-state creep behaviour of nickel can be rationalised by assuming that the modes of softening and hardening, i.e. dislocation annihilation and multiplication, are operating at rates which are balanced so that the dislocation density is constant.

'Recovery- controlled' provided that the temperature is sufficiently high, creep occurs in pure metals such as nickel by a combined climb-plus-glide mechanism – if a gliding dislocation becomes pinned, then a small amount of climb can release it, allowing further glide to occur.

Creeping above $0.6T_m$, climb occurs by lattice-diffusion (rather than, for example, by pipe diffusion along a dislocation core), and therefore this process is rate-controlling. In the steady-state regime, well-established dislocation cell sub-structures with associated sub-grain boundaries. In general, the formation of sub-grains arises principally as a result of polygonisation due to the climb of edge dislocations since cross-slip is easy

The creep behaviour of Ni

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

So, the mechanism mostly involved with the desired steady state creep at a given stress and at a given temperature level. So, for very common purposes of superalloy development, we apply actually 170 or 172 MPa stress and the first generation, second generation, third generation, or fourth generation and this generation of several superalloys has been developed in terms of a higher stress and higher creep life means time to rupture.

So, the steady state creep is one of the very important factor to look into the properties of the nickel base superalloys. So, if we see the fundamental of the steady state creep behavior, we assume that there are two different components to achieve a steady state creep, it is very simple. So, there is one mode of softening mechanism and there is one mechanism that is the hardening mechanism. And in case of all the crystalline alloys, we explain the softening mechanism in terms of annihilation and multiplication is the hardening mechanism by dislocation.

So, if one can achieve a steady state creep, it simplifies that net dislocation density in the alloy is almost constant. So, in such a case, there are some recovery control stages when temperature is very high. So, dislocation always tries to annihilate and recover the structure; however, recover from what kind of structure? So, at an intermediate temperature level such kind of polygonize network of dislocation one can see; and interaction with this dislocation with some particle will give you some effective strengthening and better creep life. Now during the recovery control stages here, the temperature is sufficiently high and creep occurred.

If you think about a pure metal let us say a pure nickel, here a combined climb plus glide mechanism is involved and if gliding dislocation pinned, then small amount of climb can release it allowing further glide to occur. So, a dislocation is gliding on a plane and climbing means that there are many planes and dislocation is climbing on other planes, and trying to find an alternative way because if dislocation encounter with a particle and if this is a ceramic particle, there is no way to cut the particle and dislocation can go to this side and this process is stopped, then dislocation should find out a alternative path and this is actually the climb mechanism.

Now, in majority of the cases you will find that the creep resistant alloys we always apply them at a temperature higher than 0.5 T_m and at 0.5 T_m or higher temperature. The diffusion is very much important process because below 0.5 T_m the grain boundary diffusion is dominating factor where as at above 0.5 T_m the grain body diffusion or let us say we call it as lattice-diffusion is important

So, now pipe diffusion is also a diffusion, which occur through the grain boundaries or along a dislocation core. Now, the process is rate controlling the lattice diffusion when we apply a temperature 0.5 T_m, means I am talking about the creep temperature. So, a steady state regime, in that case which is well established by the dislocation cell substructure, here I have shown you a dislocation cell substructure where as this is a core mechanism and the lattice diffusion always allow the dislocation to bypass. And in general the formation of such kind of sub-cell and sub-grain type of microstructure that arises due to the polygonization are due to the climbing of the edge dislocation since cross slip become easy at a higher temperature. A cross slip means dislocation basically bypasses such kind of particles.

So, if we think about TD nickel, then we can easily understand now by these total discussions I made so far that why TD nickel is one of the very good creep resistance alloy compared to a pure nickel.

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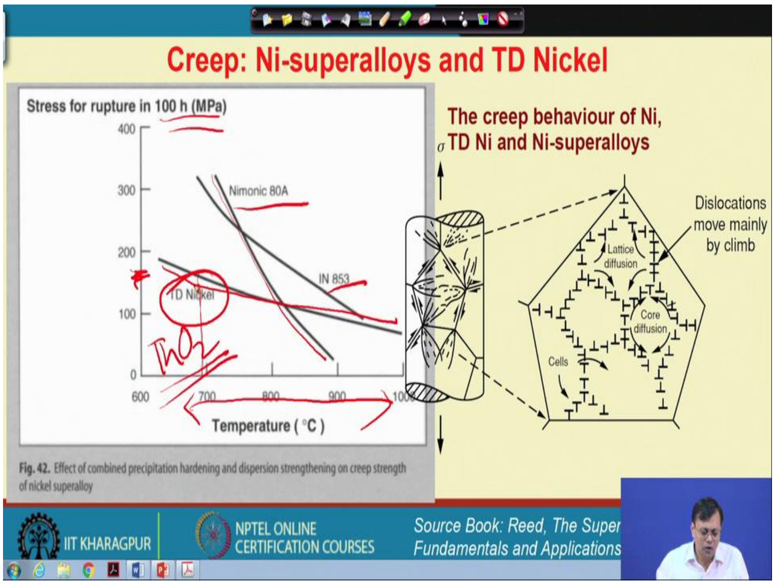

Creep: Ni-superalloys and TD Nickel

The creep behaviour of Ni, TD Ni and Ni-superalloys

Dislocations move mainly by climb

Fig. 42. Effect of combined precipitation hardening and dispersion strengthening on creep strength of nickel superalloy

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Source Book: Reed, The Super Fundamentals and Applications

Now, if I look at the creep properties of a TD nickel, if you look at the properties of a TD nickel compared to other superalloys, TD nickel since it has a nickel matrix and I told you earlier that the dispersion strengthening, here we have thoria a particle. So, this is a dispersion strengthen alloy, not precipitation hardening alloy, precipitation hardening alloys are γ'-phase.

Now, this TD nickel shows a very good creep resistance compared to other nickel base alloys, when we have a wide range of temperature of application. However, the precipitation hardened alloy, the stress for rupture basically decreases when you consider let us a here this schematic for 100 hours of use. So, we can think about for 100 hours of service life we have to apply TD nickel below 200 MPa, if we go for 700 °C. So, from this plot we understand that a dispersion strengthen alloy sometimes shows us some good properties at high temperature creep.

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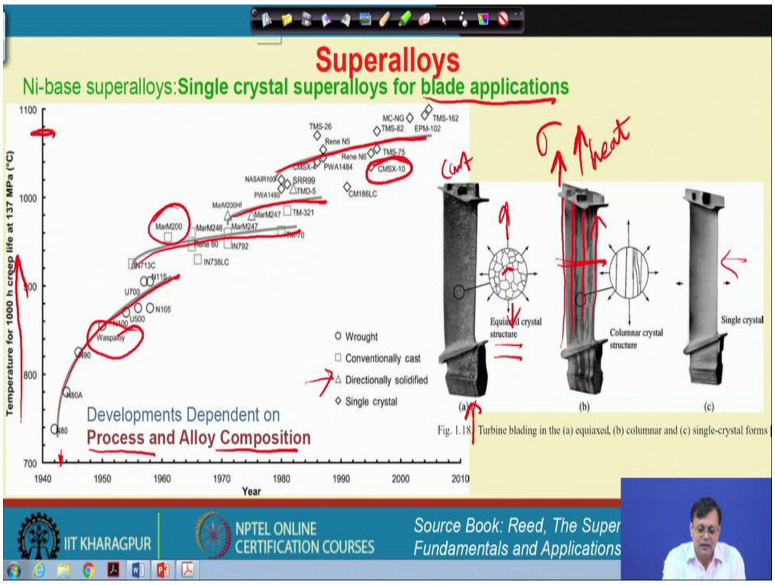

Superalloys

Ni-base superalloys: Single crystal superalloys for blade applications

Developments Dependent on
Process and Alloy Composition

Fig. 1.18 Turbine blading in the (a) equiaxed, (b) columnar and (c) single-crystal forms

Source Book: Reed, The Super
Fundamentals and Applications

Now, if we look at the very specific application of nickel superalloys for let us the blade application. With time there is chronological order of development in terms of process and composition. So, from 1945 onwards, the wrought alloys were developed. Waspaloy was one of the very famous alloys. After that, we find that the conventional cast alloy was developed, where some of these Mar series was developed. And a typical cast microstructure is shown here, this is a very common let us say polycrystalline cast alloy, where we will get a equiaxed grain structure, but when we apply stress the cracking always tends to occur around this grain boundary.

So, that is one of the disadvantage of the equiaxed crystal structure, whereas later on the directionally solidified alloys or this is a processing technique where grains are elongated along the direction of the heat extraction. So, let us say this is the heat extraction direction and the grains are also elongated along this direction. So, here you will see that if we apply some stress, the grain boundary along the perpendicular direction to the application of the stress is relatively low. And then to develop the superalloys for further higher temperature application for this temperature for 1000 hours of creep life.

So, single crystal is preferred. Here CMSX series is very much famous. So, this is a single crystal alloy and this is the finished blade and you cannot see such kind of structure macroscopically. So, there is a chronological order of development only because we need a higher temperature of application, and then we have seen that the thermal barrier coat was widely applied on this blades.

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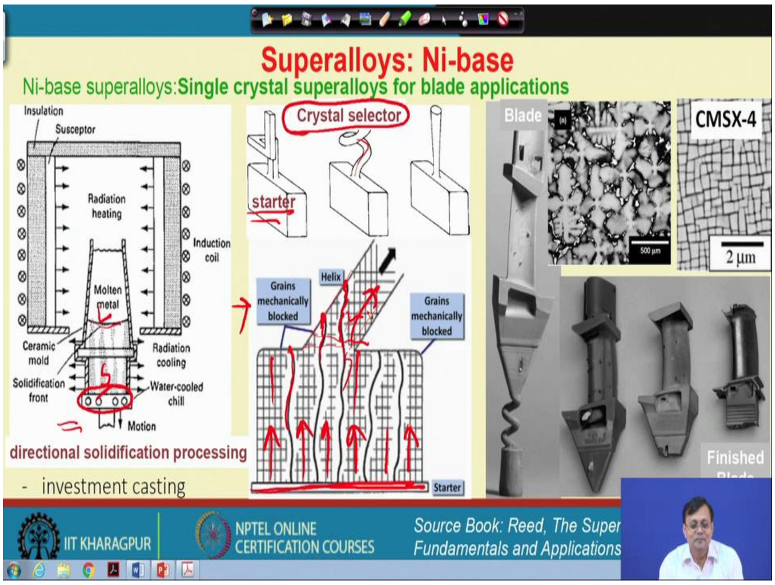

Superalloys: Ni-base

Ni-base superalloys: Single crystal superalloys for blade applications

directional solidification processing

- investment casting

Source Book: Reed, The Super Fundamentals and Applications

Now, let us think about what are the processing condition, why we really prefer such kind of a technique of producing this blades.

So, for producing blades, the investment casting is one of the very well known technique. Here we make investment slurry and we produce the mould and then we pour the liquid into that portion in the mould cavity. So, in case of pouring, if we need to grow along a particular heat extraction direction then Bridgeman type of technique, which is a slowly removal of a mould inside a heat source. So, here this is let us say a motion of the mould along the bottom direction where the furnace is fixed or otherwise means we can move the furnace on the top side.

Now, you can see that this bottom part which is a water cool chill. So, the solidification will begin in this portion first and now the grains will grow. Now you can always ask why the grains are growing only in this direction? The grain or the dendrite also form along this direction, but the growth rate means the velocity of the growth is higher along this direction. So, the growth of an inclined dendrite will always be suppressed. So, we can always get an elongated grain and slowly you remove and this is let us say a solidification front where we have liquid and we have liquid here and we have solid here. So, this is an interphase.

So, we slowly drag these mould down in order to preferably produce this directional solidification. But few seconds ago, I just said that the single crystal blades are mostly important and it resist much higher temperature and therefore, we can adopt very similar technique; however, we need to adopt a crystal selector at the bottom part of the mould. What we do here? We have a starter and then we use a crystal selector. What it does actually it mechanically block the grain, how? So, let us say this is the starter block and the grains are growing along the heat extraction direction. So, they all are growing.

Now, they have grown and here let us say I have a liquid and then I preferentially allow and drag this mould down side. So, there is the inter phase of liquid and solid. So, these are the grain boundaries and they are blocked here where this is a grain boundary which went up to here, but this is also a grain boundary which went up to here, but ultimately you will see that only this grain has a chance to grow. So, this is the only grain that has a chance to grow now it grows and then it enters into the mould cavity.

So, we start with a single grain, which fill up the whole mould cavity in order to produce this single crystal blade. This is very interesting phenomena and there are many different type of helical type of shape or this type and this a conical type of shape, there are many shapes and this is a directly cast solidified blade with such kind of helical crystal selector.

Now, we need to go for various technique and final heat treatment. So, that we can get the finished blade which looks like actually such an image and if you look at a microstructure, then you will see such kind of dendritic microstructure and after proper treatment or heat treatment we will have such a such a microstructure with γ'-phase, this is a single crystal, which CMSX 4, and γ'-phase in this kind of dark region which is the nickel matrix. So, we understood that a processing condition itself is very much essential for developing these blade nickel superalloys.

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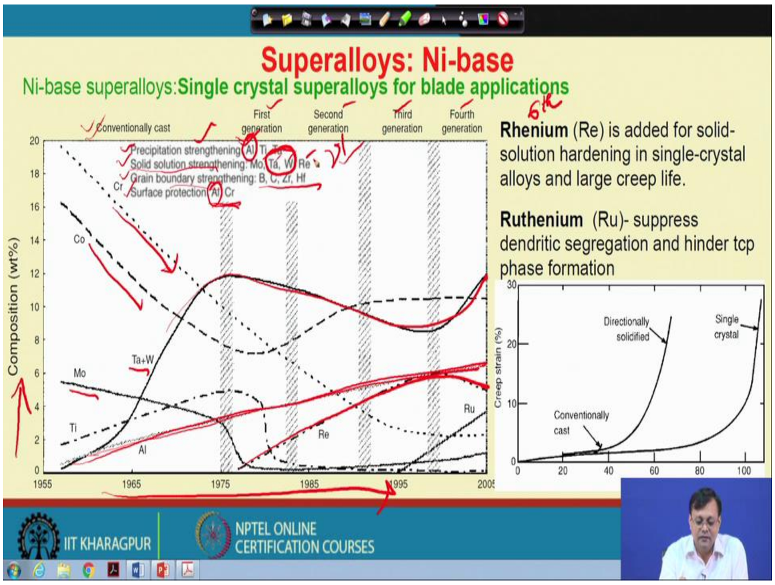

Superalloys: Ni-base

Ni-base superalloys:**Single crystal superalloys for blade applications**

Rhenium (Re) is added for solid-solution hardening in single-crystal alloys and large creep life.

Ruthenium (Ru)- suppress dendritic segregation and hinder tcp phase formation

Now, with time there are many a compositional changes and let us say the processing technique has been evolved which I just discussed few minutes ago, and here is shown one of the compositional changes that occurred with the time.

So, we already know that for surface protection, we use aluminum, chromium; however, for solid solution strengthening we basically use molybdenum, tantalum, tungsten, rhenium and for grain boundary strengthening we use boron and boride and carbide and this. So, aluminum is preferentially added also for precipitation strengthening of the γ´ phase. With time you will see that the aluminium content is increasing.

So, this was a conventionally cast which was a very old technique and then the blades with the first, second, third, fourth. Even today there are six generation blades and so, the aluminum concentration is increasing there are two purpose of this aluminums. One is surface protection, but we preferentially like that addition and increase of the γ´ volume fraction.

Now, you can have a look that the cobalt content and chromium content they also decrease and because we need to apply for higher temperature. So, chromium oxide does not provide proper protection; however, the molybdenum and tantalum they are also used, but tantalum and tungsten which is used a lot these days and the specific purpose is basically to have a better solid solution strengthening as well as the creep life and similar way the rhenium is also used for this kind of better creep life.

Now there is another element that has been added and the percentage is increasing for some specific purpose, which is ruthenium. The ruthenium basically suppress the dendritic segregation means the alloy preferably try to segregate in the entire dendritic region and it should have required hindering of the topologically close pack phases, and the effect you can see very clearly that if we have time to rupture and creep strain then conventionally cast alloy does not perform well as the single crystal blade.

So, single crystal is always better and gives us a larger time to ruptured and it require higher creep strain and so, the single crystal blades are always preferred which can be observed in this plots.

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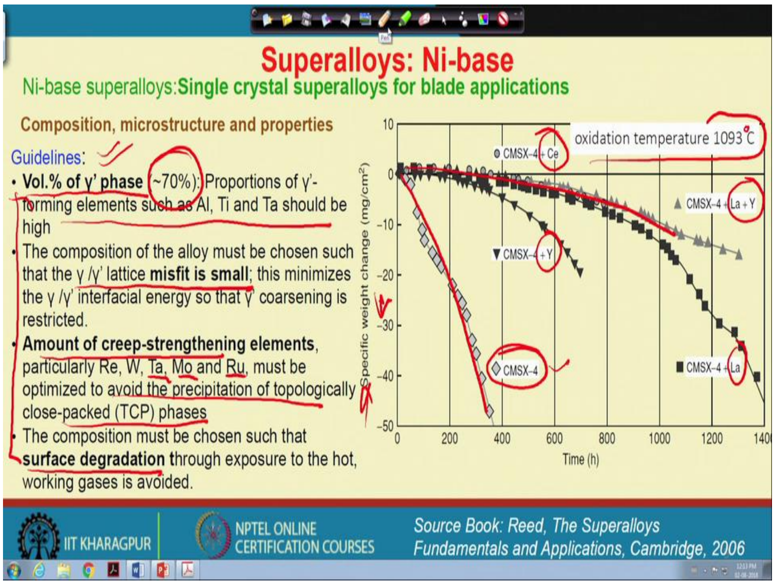

Superalloys: Ni-base

Ni-base superalloys: **Single crystal superalloys for blade applications**

Composition, microstructure and properties

Guidelines:

- **Vol.% of γ' phase** (~70%): Proportions of γ'-forming elements such as Al, Ti and Ta should be high
- The composition of the alloy must be chosen such that the γ /γ' lattice **misfit is small**; this minimizes the γ /γ' interfacial energy so that γ' coarsening is restricted.
- **Amount of creep-strengthening elements**, particularly Re, W, Ta, Mo and Ru, must be optimized to avoid the precipitation of topologically close-packed (TCP) phases
- The composition must be chosen such that **surface degradation** through exposure to the hot, working gases is avoided.

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

Now, if you think about some of the composition microstructure and properties of these superalloys the volume fraction of γ-phase should be higher like 70 % and the portion of γ' forming element that should be high. So, there are some design consideration. So, I am just talking about the design consideration. However, the misfit should be small. So, that we will get effective strengthening

Now, the creep strengthening element like molybdenum, tantalum, ruthenium that I just said and avoid the precipitation of topological close packed phases should be considered. On the other hand, the surface degradation like exposure to hot and very aggressive environment should also be considered.

So, these are the four design consideration that one must think about. Now I show you a simple example of a weight change means the corrosion rate or high temperature oxidation rate in case of a single crystal superalloy. Here you see there is a large chance of an oxidation and there is high rate of weight loss, this is a negative side. So, weight is losing this side, however, if we specifically add very small amount of yttrium, lanthanum, or let us say some rare earth cerium then we will increase their creep life.

So, the oxidation temperature here was 1093 °C. So, we must consider some of these design criteria to select what are the alloy composition. One must look at and it is for sure that.

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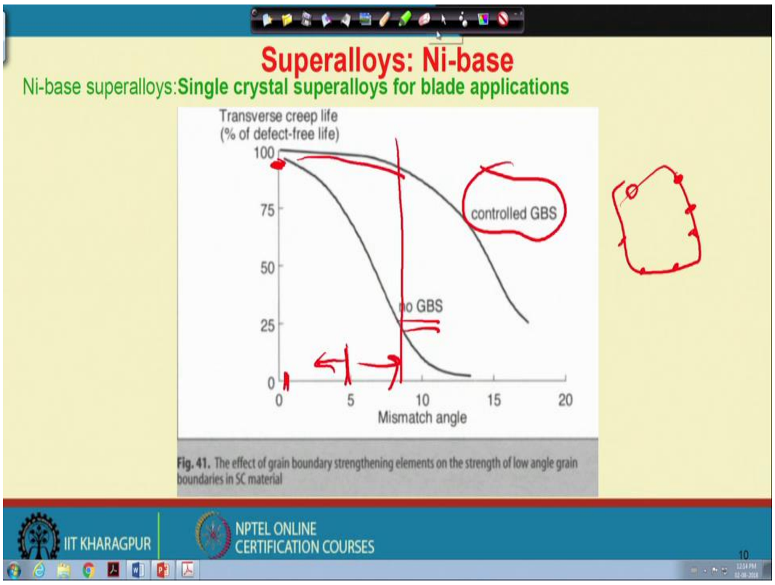

Superalloys: Ni-base

Ni-base superalloys: **Single crystal superalloys for blade applications**

Fig. 41. The effect of grain boundary strengthening elements on the strength of low angle grain boundaries in SC material

If we think about a good grain boundary strengthening, then there if there is no carbide and boride then we will get definitely a creep life which is good when the mismatch angle is less. So, less than 5° mismatch angle is always a preferable, but we can still increase it, when we have some control grain boundary strengthening. So, the grain boundary strengthening means that I have some grains and then I introduce some carbides or borides. This is always preferred microstructure for a better creep life.

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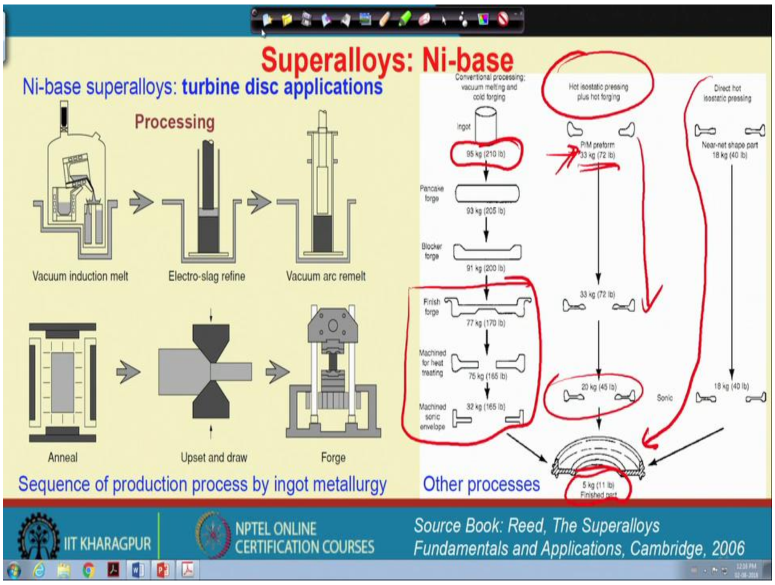

Superalloys: Ni-base

Ni-base superalloys: **turbine disc applications**

Sequence of production process by ingot metallurgy

Other processes

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

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Now, for a disk application, we should have a look at the different processing technique. To make a disk, we have to go for some particular shape which appear like this is a section. So, we start with Vacuum Induction melting then we go for some Electro slag refining process. So, that we remove some of the slag and make a purer alloy then we go for some vacuum arc re-melting so that any kind of dissolved gases to be came out after that we go for some heat treatment.

So, after this process, we come here and then we simply Upset and Draw it and then we basically forge it. This is a very conventional production process using ingot metallurgy which I have discussed for making such kind of disk; however, please have a look that in 95 kg we have to start with in order to achieve 5 kg of the product.

So, there are some alternative technique that is called hot isostatic pressing means this is powered metallurgy processed 33 kg size metal and then we simply give some pressing. So, that we get this final product. On the other hand, we can also go for some direct hot isostatic pressing in order to achieve this product; however, since these are the forge product you can easily understand that, they are not all single crystal. So, these are the polycrystalline alloys. Now here to we have some design considerations.

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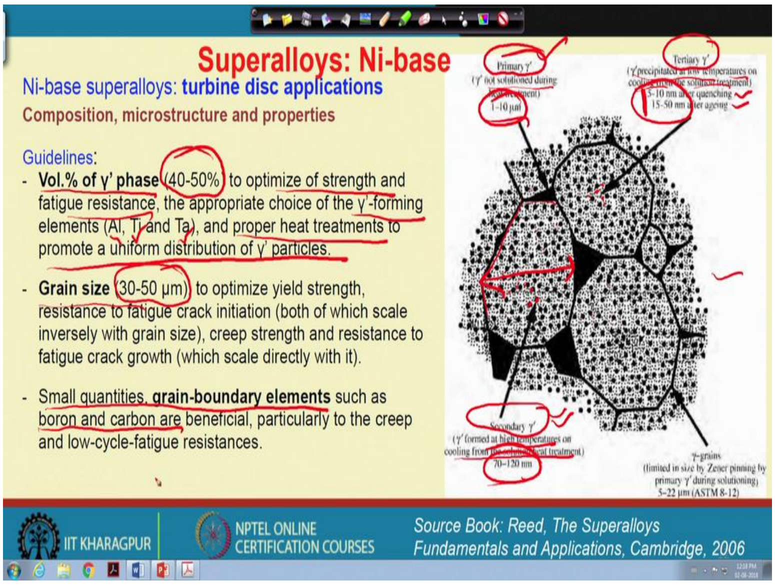

Superalloys: Ni-base

Ni-base superalloys: **turbine disc applications**

Composition, microstructure and properties

Guidelines:

- **Vol.% of γ' phase** (40-50%) to optimize of strength and fatigue resistance, the appropriate choice of the γ'-forming elements (Al, Ti and Ta), and proper heat treatments to promote a uniform distribution of γ' particles.
- **Grain size** (30-50 μm) to optimize yield strength, resistance to fatigue crack initiation (both of which scale inversely with grain size), creep strength and resistance to fatigue crack growth (which scale directly with it).
- Small quantities, **grain-boundary elements** such as boron and carbon are beneficial, particularly to the creep and low-cycle-fatigue resistances.

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

So, design consideration includes that the volume fraction of the γ′ phase should be 40 to 50%.

So, that we optimize the strength and the fatigue resistance. So, γ′ forming elements means these aluminum, titanium, and tantalum and we should go for some proper heat treatment so that uniform distribution of γ particle should be here. This is a schematic microstructure where you can see that there is uniform distribution and these are the grain boundaries.

And this γ′ phase may also appear in the grain boundary region and these are called the primary γ′. When it appears? It basically appears not during solution treatment, but simply comes during solidification itself. However, there are some secondary γ′ which form at higher temperature during cooling from the solution treatment.

So, inside if we make some solution treatment and then cool it down we can get actually this secondary γ′ phase. However, this γ′ precipitate at low temperature during cooling from the solution treatment has a much smaller length scale from 5 to 10 nm size.

So, after quenching and after ageing treatment we can get 5 to 10 nm size particle, these gives a very beautiful and effective strengthening. So, you can see that there are so many different types of γ′ phase that form in different length scale from 1 to 10 μm from 70 to 120 μmand to 5 to 10 nm size.

On the other hand, to optimize the properties we need a grain size of the matrix and let us say the grain size of this precipitate properly. So, 30 to 50 μm grain size is always preferred for this purpose. On the other hand, small quantities of grain boundary elements like boron, carbon they could be beneficial for a better creep and low cycle fatigue life. So, with this we finished today’s discussion and we will further discuss other different types of superalloys in the next class.

Thank you.