# **Principles of Physical Metallurgy Prof. R. N. Ghosh Department of Metallurgical and Materials Engineering Indian Institute of Technology, Kharagpur**

**Lecture No. # 29 Precipitation of Solid Solution**

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Good morning, today we begin a new chapter that's on precipitation from solid solution. Now, by controlling the precipitation process from solid solution, you can control the micro structure as well as properties of the material, and may be go to this display here. Now, under this we will look at terminal solid solution.

We will look at thermodynamics and kinetics of the process of the precipitation, we will learn about spinodal decomposition, we will look at homogeneous and heterogeneous nucleation, we will also learn about the types of precipitate that we come across, and when purpose of all these is to get some additional strengthening in the material. So, we will look at precipitation strengthening, the mechanisms of strengthening and we will learn about few commercial alloys.

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Now, normally if we look at solid solution the typical phase diagram looks like this, here you have liquid phase and in between this is the liquidus, this is the solidus, and here you have solid solution; there is no precipitation here this is an pure isomorphous system and if you look at the properties you will find somewhere intermediate alloy, we will have maximum strength.

So, that means any alloy addition that you add, add element B to A in that case strength of A increases; similarly, if you add A to pure B strength of B increases, and normally it is find that is some relationship between the strength the material an its melting point higher the melting point strength at room temperature is higher. So, that is why it is shown like this, and this is the optimum composition where you have the maximum strength.

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Now, what happens if you have a two phase structure? Now, within the terminal region of that is solid solution here both here and here the strength will increase. So, this is the contribution of solid solution and in between what you have which check of two phases alpha and beta, and within this region the process of strengthening is one of you can say something like it composite strengthening, so if strength increases from here to here like this.

So, if you have that strength increases if beta is stronger than within increasing volume fraction of beta strength of the alloy increases. Now, we will see what happens if you are able to control that nature and distribution of precipitate in a terminal solid solution where the solubility in this particular case the phase diagram, its show the solubility this is the solidus line here solubility is independent of temperature Now, suppose this plot would have been like this, then how could we able to control the structure or how does the structure evolves in the terminal solid solution and that is what we are going to look at today or may be another class.

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Now, let us look at a terminal solid solution the diagram is shown over here, and in this case a if you try and look at this diagram, here you have liquid this is the two phase region alpha plus liquid. Now, you have an alloy if you are cooling it very slowly what you have in between you have pure single phase alpha. So, that means you only have the green boundaries of alpha you have this green boundaries of alpha and the micro structure.

Now, what happens when you temperature goes below this solubility limit. Now, here this stable structure is alpha plus beta. Now, beta will try to nucleate and we will see where will it nucleate, if it nucleate somewhere here you will have a new surface being created and this surface will have an energy associated with it this energy is this; whereas, the extent of super cooling when it comes over here, you can say this is the driving force the super cooling is the driving force for the reaction, and the total energy change at any particular temperature,

What you can write and equation something like this delta G, this will be the volume of the precipitate, if it is sphere its volume is 4 pi by 3 r cube there will be this is the free energy of transformation per unit volume. And this is usually negative because here thermodynamically this is more stable where as surface energy this is the surface area gamma. So, usually what happens this energy is positive so right over here you find the net free energy is positive it is because right over here at the solves temperature that

means at solves delta G U this is equal to zero; therefore, total free energy changes positive.

So, as you cool here the transformation is not likely to take place as the temperature goes down there is a positive I mean there is a driving force there is a negative free energy and at certain temperature you will find this becomes zero. So, once this super cooling exceeds a particular critical value then the process of precipitation will continue Now, when we write this equation we are making some assumption that here that what you get the precipitate that forms beta the volume there is no change in the density, so density that is rho of alpha is equal to rho of beta.

So, in that case what will happen there is no volume change, but usually this is really satisfied there will be some volume change and there will be some strain energy because outside also is a solid this is a solid this side is solid it is there is a slide difference between solidification and precipitation. Now, when we look at solidification there also we applied the same principle to explain why super cooling is necessary for solidification, but in this case there is a slide difference when a precipitated forms actually it will not be allowed if its has lower density it will not be allowed to expand.

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# **Historical perspective**

Interest in precipitation process is derived from its effect on strengthening of alloys. Discovered accidentally by Wilm while studying solid solution strengthening of Al-Cu alloy by adding Mg in 1906. The strength of a guenched alloy was found to increase with ageing. Duralumin was the first commercial alloy produced in 1909. 3-4.5Cu 0.4-1Mg 0-0.7Mn + impurities 0.4-1Fe 0.3-0.6Si. Merica was first to suggest in 1920 that increase in strength is due to sub-microscopic precipitates below the solvus temperature. This was conclusively proved in 1950s with the help of Electron microscope. **NPTEL** 

So, how do we take this into count that is the key to the process of strengthening that result from this precipitation, Now, if we look at the history, so there is if you look at the slide here on the computer this is the historical perspective which is given the interest in precipitation process is derived primarily from its effect on strengthening of alloys, and this was discovered like most discovered is of alloy; that it discovered accidentally by Wilm while why he was studying the solid solution strengthening of aluminium copper alloy by adding magnesium in 1906.

In fact that aluminium the discovering that is bulk it was available and bulk towards the end of 18th century and during that time you know, that aviation also there was keen interest and developing aircraft and aluminium happen to be a very light alloy, and it was a that this can help promote or help in the design or new generation of aircraft. So, there was a lot of interesting improving the strength of aluminium alloy and their process of strengthening that was known then was a solid solution.

And when looked at some of these alloys he found if you quench the purpose of quenching was by quench you can separate precipitation, and then you can keep more amount of magnesium in the solid solution, and logic was if you keep more amount of alloy element in the solid solution the strength will increase, but in his case he found that there was no increase in strength but the quenched alloy it was left you know, after quenching for few days. And then suddenly, one day he tries to find out the hardness of that he found the hardness increases an increases with the time of ageing, this is how the commercial precipitation hardening alloy was discovered and duralumin was the first commercial alloy which was produced in 1909, and this is the composition it had a 3 to 4 percent or around 4 percent copper.

It had say some amount of magnesium little bit of manganese, and some impurities some amount of iron and silicon, and these are all in small amounts and people wondered why does this strength increases at that time to look at the micro structure only optical microscope was available. Now, America was the first to suggest in 1920 that increase in strength is probably due to some sub microscope precipitate which form below the solves temperature.

But this was proved at this was only a  $(())$  he just put this as a postulate, and this was conclusively proved only with the help of electron microscope when it was a available and the technique of sample preparation, and examination of sample and transmission electron microscope was possible. And this was this happened in 1950s, and there after they has been considerable what in this area, and we will see how we can control the process of precipitation in these solid solutions this unstable solid solution that term stable terminal solid solution to get the maximum amount of strengthening. And we will see how that extend of the strengthening, they will be control by controlling the phase transformation or precipitation process in this alloy.



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Now, let us look at the terminal solid solution here, and what happens? If you take this, and if you heat it say suppose take this alloy and at room temperature, suppose this is its micro structure, you have this area is say if you take a supersaturated solid solution. And look at this is the micro structure at room temperature here, this is the alpha and these are the precipitate beta, this is the equilibrium structure at this temperature.

Now, suppose you heat it to this point. Now, what happens if you keep it for some time the beta will dissolve because of phase diagram, it say that this is the region where same alloy should exist in a single phase form and $\ldots$  Now, say suppose you cool it very fast. So, that means you have heated heat here, this is the solves this temperature and soaking it for some amount of time, and then quench in water or cool very rapidly like this, then it is every there is every likelihood that you will get a supersaturated solid solution.

So, what does supersaturated means it means by force you are keeping a large amount of solid in this structure is something like this, this is the excess amount this is the equilibrium this is the solubility limit at room temperature, let us say and by force we are keeping this is the amount of solute. So, that means in terms of excess solute percent we can say this is the excess solid which is excess solute atom which is percent in the solid solution so this is a major instability you can say and other way we can say we have a super cool system and this is the temperature difference, so this you can see is a major of instability. Now, this process of taking a forming a supersaturated solid solution it is known as solutionizing heat treatment, so here you soaking for giving solutionizing and then you have quenching and by this process it is possible to get an unstable solid solution.

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And now what happens? We will see how we can control the process of precipitation process of precipitation and supersaturated solid solution. Now, if we look at the freeendecalp the free energy composition diagram somewhere here, let us say when you have say at this particular temperature may be it is easier to explain, this is the equilibrium structure and we have a supersaturated solid. Now, let us say the free energy composition diagram of that supersaturated solid solution is given by this curve.

So, this is the free energy and this is composition that is atom fraction B this direction Now, this is the solubility of B in alpha at room temperature. Now, here we have kept this much of excess, so this is the amount of solute in alpha prime, we call this supersaturated solid solution as alpha prime. Now, for alpha prime its free energy is this so what you have this is the free energy difference, so its stable structure it is made up of this is the composition of atom fraction B in alpha this is the atom fraction of B in beta.

So these two this is mixture of alpha, and beta is the equilibrium structure at this particular temperature and here by force we have kept this at room temperature.

So, there is a major of some amount of instability and this delta G we call this as delta G v this is the major of instability, and this is negative at room temperature, so there is a driving force, but why it was if there is... And it is quite large, if this is the temperature difference from this room temperature if this is the temperature difference it is quite large why does not the precipitation take place and this is.

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To understand that let us look at this diagram. Let us consider we define the driving force that free energy delta G v is the driving force for precipitation and this can be major either in terms of this extent of super cooling that is the delta T, so this is the temperature from which it has been quenched. And after quenching let us say we got this supersaturated solid solution and then we keep it at a particular temperature here, and leave it at that temperature. What will happen to the micro structure of the alloy? And to understand that, so let us see.

So, this is at this temperature this is the solubility, so what we can see under this condition you have either this you can see this is the driving force or this is a major of the driving force, and they can be converted to free energy that is delta G v and this is negative, but here precipitation does not take place there is something like this. This is the supersaturated solid solution, and this is the equilibrium structure, there is this is

negative this as lower temperature, but always any phase transformation of from kinetic point of view, we know that unless it covers this energy hump.

So, there is an activation hill this must be over come for the process to continue precipitation process, we will see the origin of this activation hill why do we have this activation hill, but it is very easy to follow that the micro structure optical micro structure examination. We will know, show any sign of precipitation, but you can follow the transformation by measuring hardness this is the weakest hardness, and if you measure the weakness hardness at this particular temperature after ageing for at different length of time at this temperature. Then you quench it to room temperature and measure the hardness you may find, if this we your as quenched hardness, and if you age you will find this hardness goes up goes passes through a peak and then comes down.

Similarly, if you change the temperature of ageing, we will find say if you age it at a even lower temperature then you find you get even a higher increasing hardness. Now, we will try and see that means effect of a increasing temperature is that hardness peak increases, hardness increases peak hardness say hardness this is peak hardness this increases as ageing temperature decreases, and the time at which the peak hardness is reached this is lower higher the temperature. That means the process of a precipitation is faster at higher temperature; therefore, the peak is attain faster, but therefore to get the full benefit you have to optimize, you have to optimize the ageing time and temperature.

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so that you get an optimum strength which is usable which has the proper application. Now, let us see the process of precipitate formation in a little more detailed. Now, let us look at first homogeneous nucleation, so assume a precipitate is forming and which I just mention right in the beginning of the class this is an alpha green, and here you have a precipitate forming.

So, process of formation or precipitated is creation of a new surface, and let us see alpha beta is the surface energy per unit surface and here we mention that the total free energy change will be some of these two and process will spontaneous only if this free energy change is negative. Now, we mention that you get a proper expression for this we must look at the contribution of strain energy which is very important the precipitate that forms may have a different density.

So, there will be some amount of volume change, and this volume change results in additional stored energy, and this stored energy you can say this is the strain energy which is proportional to volume. Therefore, the total free energy change apart from the volume free energy change we must add this strain energy component, and for the precipitation process to take place this total free energy; that is total free energy should be negative for the process to containing.





Now, let us see look at the classical nucleation theory. Now, there is a one point as where as we go through we will find that when we look at the precipitated in an optical microscope, we see that after ageing there is no precipitate, so that mean precipitates are extremely fine. And when it was possible to see them under electron microscope they were of a much smaller dimension something like may be 50 hamstrung and nano meter dimension.

So, therefore, people question that at that level will classical nucleation theory the applicable, because at smaller particle that is more lightly that they will follow quantine mechanics, but yet many of this process of precipitation process that phase transformation kinetics whatever that the experimental strain that, we see can be very well explain in spite of this conceptual that limitation the precipitates sizes very small. Yet most of the features that we observe under experimental condition in number of alloys systems the classical nucleation theory, and explain that very well.

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Now, a quick look at this if we see that if r is the radius of the nucleus this is the bulk free energy per unit volume this is the gamma, and you can quickly write this and I will leave to you because it is exactly the same procedure. If you look at if you differentiate this total free energy at a particular temperature is a function of the size of the nucleus and what will be at stable nucleus how will you find this stable nucleus?

So, what you do you differentiate this equated to zero, if you do that and we collect algebric simplification you can derived easily, and it can show that critical nucleus size is twice gamma that is surface energy gamma alpha beta over the sum of the free energy by free energy that transformation plus the strain energy contribution. And similarly, this is the energy harm this is the magnitude of the activation hill we can say, and I will leave it you how you will be able to these are the very simple derivation we have done it similar derivation during solidification.

Now, if you look at this critically what is important to record nice that this is negative and this is positive so what will happen, so here r star that critical nucleus size what will happen if that strain energy increases, if strain energy increases. In that case this denominate a magnitude of the denominate will become smaller magnitude, because this is negative the magnitude will become smaller the r star will be larger, so what it means is that the critical nucleus size will increase.

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So, which is shown over here as the strain energy increases this delta G star that activation hill will increase the critical nucleus size will increase, so that means the process of you can say the precipitation will become more difficult. If this strain energy there is a high energy of a high amount of a strain energy as a result of precipitation, if that strain barrier is to over come you need to give more amount of thermal activation.

So, that means in a way, and in another way if other way if you look it, if you can play with this surface energy, if gamma decreases then what happens then critical nucleus size also decreases. So, that means you can by playing that with alloy addition or alloy design you can play with this possible to change both this surface energy as well as this strain energy and by controlling the it is possible that we can promote homogeneous nucleation; homogeneous nucleation will be promoted when it will be promoted, if gamma is very small and E s the strain energy is also very small.



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And we will see as you go proceed to the lecture when will it be possible to promote homogeneous nucleation, and why we will need homogenous nucleation is, if we want that we will see as we go through the mechanism of precipitation in any micro structure; these are the grains you have this grains. And we know that strength of these grains can be improved, if we can make the movement of this location which moves through the grain more difficult, so if you have more amount of precipitate within the grain.

So, you need to have not only precipitate and the grain boundary you need to have lot of precipitate within that grain, and how can you get; that means you have to surplus because the grain boundary provides a favourable nucleus inside, so normally precipitate will like to form here and primarily this grain corners and the grain phases. Now, to get a good precipitations strengthening you have to have nice array of precipitate within the grain, and this can be promoted by promoting homogeneous nucleation.

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And now, let us look at a little detailed the process of heterogeneous nucleation.

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Now, when this heterogeneous nucleation takes place, one of the preferred side is this is the grain, so this side is a grain alpha this is grain, and here you have this is the beta precipitate which has formed. Now, if we say that this is the angle here it substance the angle between this boundaries alpha, and beta boundary here, and so these are surface tension and at this point the force of they should be equilibrium.

So, that means this relationship to be satisfied that is gamma alpha alpha; that is force acting along this boundary this should be equal to force acting in this direction, and this is the component the horizontal component of gamma beta cos theta twice, and when a new precipitate forms here what happens in a grain boundary, you have this is the alpha this is the alpha, see if a precipitate beta forms here what happens. So, this amount of alpha alpha boundary, this appear in its place what we have we have certain amount of a alpha beta created.

So, this is one A alpha beta this is the other A alpha beta, and assuming this A to be spherical caps this is the part of a sphere and we call it this is also a part of sphere, and this is a double spherical cap double spherical cap. So, assuming then this to have this type of shape it is possible to calculate what is the increase in area of changes in area, and what is actually the change in the surface energy as a result of this type of a heterogeneous nucleation of precipitate. And you can show that under condition what will be the critical nucleus size, and if you go through the exact like the calculation procedure is exactly same. You have to considered this is the free energy of the driving force for the process of precipitation, this is the strain energy, this is the increase in surface area the twice alpha A alpha beta.

So, this is times gamma alpha beta is a increase in surface energy, and this is the surface which is lost. So, gamma A alpha alpha area is lost and this is the energy which is lost. So, in this way you can write the full expression. Now, you can also find out easily the volume of the double spherical cap which is given here the area A alpha alpha that means area of this circular shape this circle area circular area, this A alpha alpha which disappear is this, and new alpha beta area which is created this is that.

So, if you go through these steps and you can show that the heterogeneous nucleation the dimension of the expression for a heterogeneous, and homogenous nucleation will be exactly in this same. So, whether it is heterogeneous or homogeneous nucleation that dimension at that is radius of this critical nucleus is same, but that does not mean that volume of the critical key thing is the volume here is only of the nucleus is only this much only, this is the size of precipitate which needs to be created.

So, compare homogeneous nucleation that volume of that precipitate that forms is much smaller here, so that way and the heterogeneous this energy hump you will find that this energy hump is a function of this shape factor this function of theta is called as shape factor, and heterogeneous nucleation that energy hump reduces. So, that means this is for homogenous, this is for homogenous this is for heterogeneous.

So far heterogeneous process the energy hump is less so this is should be the normally the most preferred process of precipitation, and you can try and find out for different values say put different value of theta. And the shape factor and see it is also possible to make it can you make this when is this zero? I leave it to you to do it.

> **Heterogeneous nucleation** Ш  $60$ cos( Grain Grain Grain boundary edge corner

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So that means in such cases, if this is equal to zero in that case virtually there is no activation hill and a quick look at the magnitude of this surface, this energy or nature of this surface energy which is demonstrated here which is shown in this slide here. If you plot just make a plot of this gamma alpha beta against gamma alpha alpha.

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If, you plot this so when this is at theta when theta is equal to 60 degree cos theta is half, so you will find that here this ratio is one whereas, if the angle is less than 60 degree this is less than one, and also this magnitude will also depend on depend primarily depends on the shape factor or this will decide the theta this magnitude of theta or you say since theta is related to the ratio of this surface energy that will determine this shape function.

And if you look at the heterogeneous the volume of these two, you can easily show the volume of a heterogeneous that activation hill over homogeneous activation hill, this is the exactly equal to the shape factor, and this is also equal to the ratio of the volume of the critical nucleus under heterogeneous condition. And volume under homogeneous nucleation these two, and if you plot these two ratio against a function of cos theta, then if the precipitation takes place in the bulk, then this will be the nature if the precipitation takes place at the grain boundary of the grain phase.

So, that means here in that case that means the edge of the boundary then this will be the plot, and if it forms can form on the grain edges these free grains which are meeting, so this is the nucleation this is what we consider this type of precipitate, so I will call it this is one so this represents 1 this grain edge this represents this is the 2, and this is 3. So, this represents given by 3, so that means preferred side will be the grain corners heterogeneous nucleation, and in that order and in three dimension you can see that at a particular point in a 3 D, the four grains they can meet at a particular point and which is try to show and this 2 D diagram.

So, it is something like this started that is a tetra hydrol, so this is one boundary, this is another, this is the other, this is the third, and this is one of the edge as grain edge. So, this will be the grain corner that four grain corner in that 3 D will be the most important side or next is this grain edge this will represent an edge, and this is the represent grain boundary or just grain phase.

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Now, let us look at the nature of precipitate when precipitate forms, so this is the matrix when a precipitate forms, it is quiet likely that precipitate this coherent they can form as a subscript. That means the coherent matrix, that means the matrix may form as a subscript, and the beta may nucleate on this subscript in that case there is a MB likelihood; that this crystal structure will determine the nature of the precipitate and in one extreme case which is shown over here is a perfect lattice matching. That means the dimension of the lattice phasing of a particular direction here in alpha is equal to lattice dimension along a particular direction in beta, and when this happens there is a very little strain energy associated, in this case whenever we have this kind of a perfect matching, we call the precipitate to be coherent.

So, this interface is a coherent interface and this will have least energy surface energy, another case if you can say that the lattice parameter of alpha and beta they are slightly different from one another, so it is slightly that means at that interface we can visualize as if you have a edges location which is shown over here this is alpha, this is beta this dimension is a little less than this. So therefore, you have they are little this started this is the lattice that line which is showed as lattice strain, and here you can imagine there is an extra plane of atom.

So, which represents a dislocation that means the interface between these two precipitate is made up of a number of edged location, so this type of precipitate we call it partially coherent precipitate, and you can easily calculate based on the difference in this lattice parameter what is the magnitude or this is the difference in the lattice parameter delta A alpha A is the lattice parameter of alpha A beta is the lattice parameter of beta along the particular direction.

So, this is you can say a major of that lattice strain, and in this particular case A alpha lattice parameter of alpha is greater than A beta, and it is possible to find out if this is the lattice mismatch, you will find out that this mismatch is equal to that lattice parameter when this distance is a say after certain amount of distance say over this distance you can say here there is a perfect matching here there is a perfect matching, but half of this you have maximum mismatch.

So, you can say that over a you can find out that means what will be the distance between these type of dislocation grain boundary dislocation in this kind of partially coherent matrix and this distance is capital D and incoherent matrix there is no relationship between the orientation or between you can say the lattice plane here and lattice plane there, so in that case which is represented like this. So, this is an incoherent matrix, so energy wise this will have the minimum energy, this will have intermediate energy, this type of boundary will have maximum energy therefore, if we look that upon that strain energy, so this is or that gamma that concept you can say the gamma which prevents so here you can say that since gamma is less there is no surface energy vary of precipitate to form whereas, in this case you have maximum amount of a surface energy varied which must be overcome of precipitation to take place. So, this type of incoherent precipitate definitely it will take place on grain boundary grain corner or grain edges and this will have intermediate characteristic.

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Now, what type of precipitate will be interesting to look at will be ideal for precipitation hardening, Now, we are just seen to get a good precipitation hardening you must have uniform precipitate throughout the matrix and low gamma, low precipitate that boundary energy and low strain energy will promote homogeneous precipitation.

So therefore, if you can promote this in that case you are likely to get more uniform distribution of precipitate and which can give you better precipitation hardening; therefore, the condition for precipitation hardening is the most solid solution terminal solid solution where coherent precipitate can form or there is some amount of strain energy also in that case the exhibit precipitation hardening; that means by controlling alloy system which has some metastable precipitate, some coherent precipitate forming with a low surface energy, and some amount of lattice strain in that case these are the alloy which will exhibit precipitation hardening.

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So, today if you look at what we have consider is we looked at stability of supersaturated solid solution, we looked at stability of supersaturated solid solution, we also looked at homogeneous and heterogeneous precipitation process. And we have seen that if we can promote homogeneous precipitation then the process precipitate distribution is more uniform and they are likely to give better precipitation hardening, we looked at heterogeneous precipitation.

We looked at the what are the potential sights of heterogeneous precipitation then we have seen they are gradation the grain corner are the most where four grains; that means in a three D in a network there where that these are grain corners are the point where four grains they meet in a space and three dimensional space, so these are space the place is where the precipitate forms there they will be I mean minimum disturbances that are lowest that surfaces free energy change. So, they will be the most favoured site next will be the grain edge and then the again phase, and we also looked at type of the precipitate and origin of what type of precipitate what is coherent precipitate, what is semi coherent precipitate? What is incoherent precipitate?

If any may large system you have in coherent precipitate, you cannot get precipitation hardening, but if there is a chance of forming that precipitate that coherent precipitate.

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And in that case you get a precipitation hardening in fact these coherent precipitate which were first absorbed the aluminium copper along they were first shown to extend D fraction technique there are precipitate, there are strain fields associated with precipitate and this was done by Greener Preston. And therefore Greener Preston and it is known as G P zone, so these are meta stable precipitate which initially form during aging we are known as G P zone, and these are meta stable precipitate and they are free energy is higher. But because of kinetic consideration, because of that surface P energy consideration they appear before the stable precipitates appear in the matrix, we also looked at the effect of aging on hardness and strength. We also looked at if you over age again the hardness decreases, so this is the a precipitate becomes, so course we can be seen under an optical microscope when you can see precipitate under optical microscope you can say that is an over age structure it has lost its precipitation strengthening.

And we also looked at the necessary condition for precipitation hardening and in the next class, I think we will look at not only another mechanism for homogeneous nucleation, and also we will look at the mechanism or precipitation hardening, and how the precipitate distribution the size and falling fraction of precipitate determines that extend of precipitation hardening. And we will also look at little more critically on the nature of hardness versus time plot during ageing, so the next class we will take of these and thank you very much.