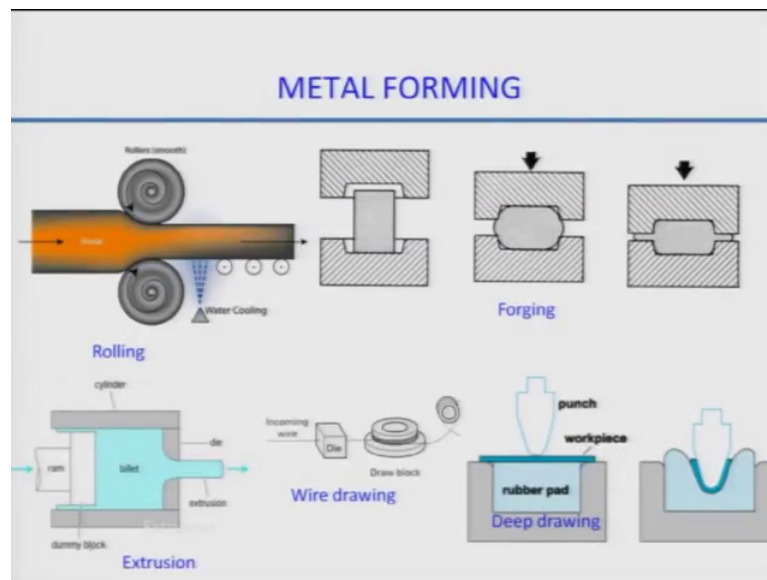


Phase Transformation in Materials
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Lecture – 46
Recovery, Recrystallization and Grain Growth

So we are going to start a new topics today that is on to recovery recrystallization and grain growth this is a different topics from whatever we have discussed earlier we called recovery recrystallization can roll as a phase transformation because new kind of grains and phases can form from our old or parent phase that is why it is called a phase transformations. But the phase transformation is different here in the sense that this is mainly due to the defect structure in the material. So, therefore, before we go into these things we should first discuss how actually defect structure form during deformation of material, you know most of the materials are basically prepared in this world is the final form after different types of deformations.

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So, we use techniques which allow us to prepare these final products they can be either rolling or forging extrusion wire drawing or even deep drawing and many other things I am not discussing all of them, but these are the main processing techniques which are used to prepare objects which you see the real life follow in the final form like if you consider the fan blades they are prepared from a steel after severe plastic lot of plastic

deformation using rolling similarly many other objects. So, in each of the deformation processes which will be discussed in 7 other courses materials are subjected to plastic deformation and plastic deformation means; obviously, dislocations, point defects and twinning these are the main modes of deformations therefore, defect structures will be formed in the material by these process.

Now, once a material is subjected to this kind of metalworking what is or metal forming by application of the force lot of stress is generated. So, thus they are annealed subsequently; that means, they heated to a high temperature to remove these stresses or rather to have certain kind of equilibrium situation in terms of the defects in the material although it is not a true equilibrium, but some sort of a adjustment in the structure defect structure of the material after the work metal forming is done. How these are done that is the main topics.

So, working or forming can be done in either by hot working like a temperature higher than about 0.5 or melting temperature of the material like for steel suppose many types of steel is about 15 to 600 degree Celsius temperature. So, any temperature pure 800 is known as hot working for the steels so that means, steel will be heated to that temperature that is about 900 to 1000 degree Celsius temperature and then deformed.

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Hot Working

- Deformation at temperatures above *recrystallization temperature*
- Recrystallization temperature = about one-half of melting point on absolute scale
 - 1 In practice, hot working usually performed somewhat above $0.5T_m$
 - 2 Metal continues to soften as temperature increases above $0.5T_m$, enhancing advantage of hot working above this level

So, normally this is done because at high temperature the strength of the material decreases substantially. So, that way it is easy to deform; obviously, ductility increases

ductility will be quite good even have room temperature. But high temperature it will be substantially increased and this allows us to deform the material extensively and that is the hot working is done at high temperatures similarly one can also do a cold working that is deformation of a material at a temperature slightly above slightly below not above actually below $0.3 T_m$.

So, for steels $0.3 T_m$ is something like about 500 degree Celsius temperature, 400 dcs temperature over 350 to 400 dcs temperature is what is known as cold working people steal. So, many cold working processes are being important for mass productions normally cold workings are more prefer. Nowadays because we have minimum and no machining requires subsequently. So, these possesses that is why cold working near nets a processes

So, that is why. Nowadays in any steel plant if you visit there will be cold rolling mills; that means, the steels are deform at very very low temperature very very low means compared to the merely temperature of a steel something like 300 400 or even lower to produce products final products. So, thus there is no subsequent of persons will be required in hot working temperatures are high.

So, therefore, steel will get oxidized and. Now side scales forms thus requires subsequent secondary operations to remove them or to get the final shapes using machining or some other processes, but in cold work you do not need that and that is why this technique is widely used. Nowadays in the steel plants even that is true for many other metals also some metals it is better to use something known as warm working not like as working at a such a low temperature, but in the temperature between $0.3 T_m$ to $0.5 T_m$ materials are heated and deformed using these processing techniques a rolling forging extrusion of drawing operations.

So, what actually happened at this temperature normally as you see the lot things written there like recrystallization will not happen, but something else will happen. So, therefore, warm working is also another way of deforming a material at a temperature intermediate between cold and hot working.

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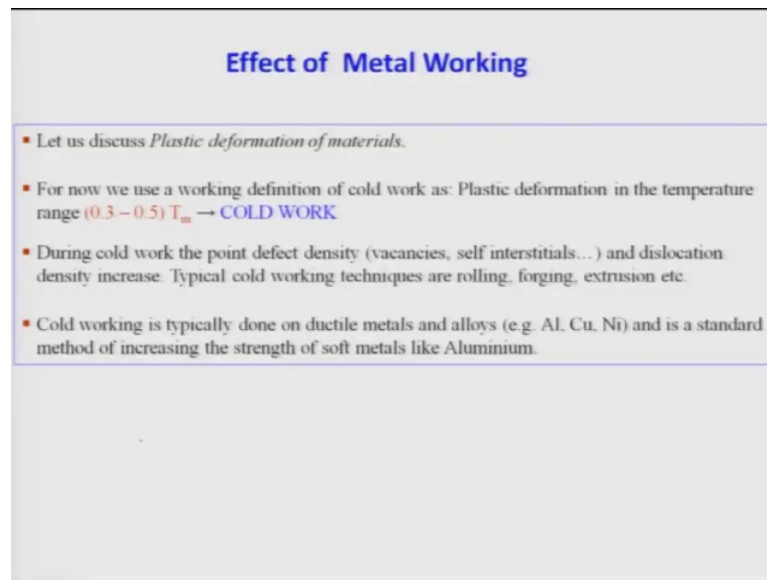
Warm Working

- Performed at temperatures above room temperature but below recrystallization temperature
- Dividing line between cold working and warm working often expressed in terms of melting point:
– $0.3T_m - 0.5 T_m$, where T_m = melting point (absolute temperature) for metal

So, question is this once we deform a material using a cold working that is what is the paramount importance for us, but many things will happen even hot and warm working also whatever discussion we are going to do. When we deform in a cold conditions that is at low temperatures or a room temperature then materials will be accumulating what is known as a defect structures as I said at the beginning. Defect structure means sale may a pack molting dislocations or point effects or twins depending on the what kind of mode of deformation happens in the material.

So, therefore, main effect of these kind of working or deformation is basically plastic deformation induced defects right.

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Effect of Metal Working

- Let us discuss *Plastic deformation of materials*.
- For now we use a working definition of cold work as: Plastic deformation in the temperature range $(0.3 - 0.5) T_m \rightarrow$ COLD WORK
- During cold work the point defect density (vacancies, self interstitials...) and dislocation density increase. Typical cold working techniques are rolling, forging, extrusion etc.
- Cold working is typically done on ductile metals and alloys (e.g. Al, Cu, Ni) and is a standard method of increasing the strength of soft metals like Aluminium.

So, we use working conditions definitions as cold work as I said which is basically or warm working is basically between point less than 0.3 or 0.3 to 0.5 T_m and we always say it is either cold work or warm work.

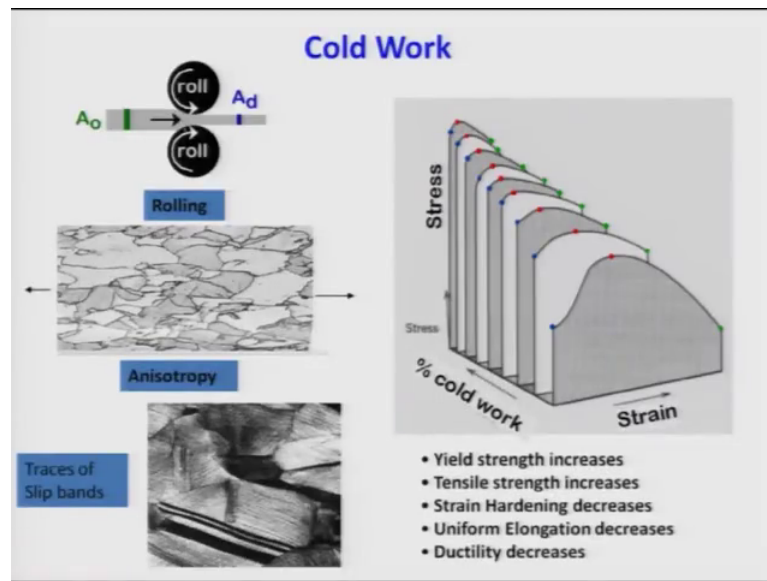
So, in both the cases a lot of defects are generated like vacancies self interstitials dislocations and this leads to decrease the ductility of the material or increase of the strength of the material. Obviously, because if there is a more number of defect structure in the material deforming the material is difficult because it requires more force to deform it further and that is what strength increases. Subsequently as there are dislocations and defect structure presents the material making it ductile is also difficult why because of the presence of these defects already in the material moving another new set of defects or dislocations are twins inside the material is more difficult because the existing defects will try to put barriers for the dislocations or the motions of the dislocations or the twins.

So, that is why cold working leads to decrease if the ductility increase in strength, but increasing the strain is because we want materials to high strength, but decrease in ductility is basically not a good signature because in the actual applications this decrease in ductility can create problem material can actually fail miserably. So, that is why all this cold work or warm work materials are subsequently annealed or subsequently heated to temperatures. So, these kind of defect structures can be anneal out or defect structure

can be rather organized properly. And that is it what that is what is happens in recovery recrystallization and grain growth these techniques actually these class of phase transformations deals with how to control the defect organization inside a cold work or warm work material.

Now, question is this as you know this has given one example, suppose if I take a steel and piece of steel and rolled between 2 rollers as you shown here.

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So, rolling will lead to deformation and materials gains will become elongated and it will create all kinds of defect structure like slip bands you can see here. This is taken from the book of callister metals engineering, as you see here mostly deformation will be isotropic why because they put the gains will be oriented perpendicular to the direction of the loading as you know loading is happening this way. So, the grains are oriented or elongated along the direction perpendicular to the direction of loading that is obvious because and the one direction the material getting compressed.

So, that is why the grains will try to elongate in the direction perpendicular to it and this defect structures place to increase in the yield strength increases tensile strength, but decrease in a uniform elongations and decreases the ductility activity. As you see here very clearly as you this is a plot between stress and strain as a same time the cold work is shown cold work means percentage deformation induced in the material it is just like that if I take a roll piece like that. So, $A_d - A_0$ divided by A_0 is what is known as cold

work that is the amount of deformation induced A_d is the final thick you know area cross sectional area A_0 is the initial cross sectional area. So, therefore, final, but initial divided by final divided by initial is what is known as the cold work and this cold work can represent terms of percentage the when you multiplied with hundred

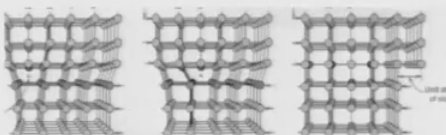
So; that means, $(A_d - A_0) / A_0 \times 100$ percent what is the percentage cold work. So, as you see here when a cold work is less you have a low strength, but huge low strain means stress is low to deform, but that, but strain is large; that means, material is ductile. As you increase the cold work subsequently the stress required to deform is increasing that I already discuss that is the main reason up for the increase of strength because you have more defects in the materials you need more stress to deform it further plastically because there are leads so many defects presents which will hinder the motion of the defects further into the material.

So, this will lead to decrease in the strain or ductility that is what happens in a real cold work situations. So, this decrease of ductility is the major problem in the further application on the material and that is why they are deformed.


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DISLOCATION MOTION

- Produces plastic deformation,
- Depends on incrementally breaking bonds.



Adapted from Fig. 7.1, Callister 6e. (Fig. 7.1 is adapted from A.G. Guy, *Essentials of Materials Science*, McGraw-Hill Book Company, New York, 1976. p. 153.)



Plastically stretched zinc single crystal.
Adapted from Fig. 7.9, Callister 6e. (Fig. 7.9 is from C.F. Elam, *The Distortion of Metal Crystals*, Oxford University Press, London, 1935.)

- If dislocations don't move, deformation doesn't happen!


Annealed material

$\rho_{\text{dislocation}} \sim (10^6 - 10^9)$

→ Cold work →

Stronger material

$\rho_{\text{dislocation}} \sim (10^{12} - 10^{14})$



Adapted from Fig. 7.8, Callister 6e.

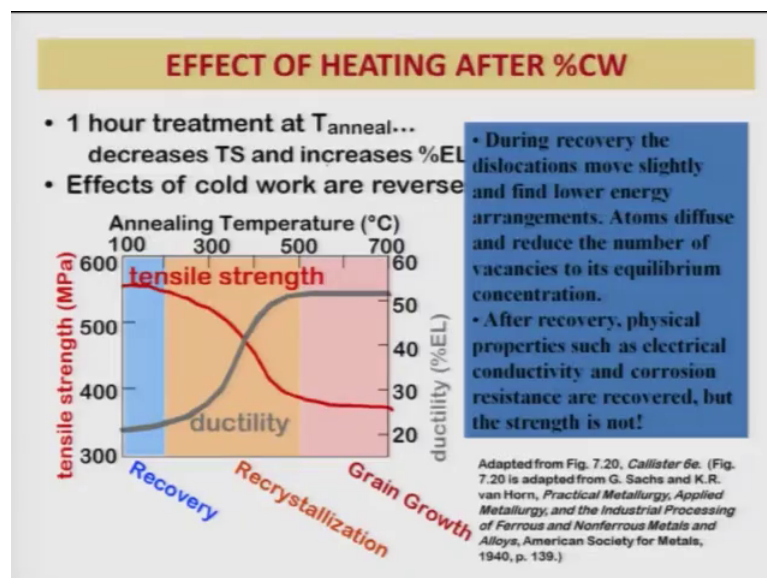
Now, you know the prominent mode of deformation plastic deformation is by dislocations and a dislocations actually move by application of the shear stress. So, if I show a 3 dimensional picture this, this is a edge dislocation this is an edge dislocation you see here that is an extra row atoms and as the shear stress is applied in this way the

dislocation moves and it reaches the surface after some time. So, dislocations if the dislocation do not move deformation does not happen as obvious or is same thing for the twin, twin is another mode of deformation for the hexagonal materials.

Now in a plastically stage zinc crystal that is what is shown here you see here that dislocation motion leads to formation this kind of slip bands which is which is formed because of dislocation motions on the surface of these crystals and they are like that So, therefore, in a normal material if we measure the dislocation densities of the obtainable 6.49 per meter square per meter actually, but on the other hand in a cold work material this can go up to several orders may should have 10 to the power 12, 10 to the power 14 dislocation density per meter. So, that is a huge number of dislocations presents inside a deformed material 9 to 14 is 5 order of magnitude higher. So, the line of our dislocation would reduce the ductility increase the strength.

Similarly, for the twins, now as you as I said to recover the ductility we need to heat it up and all other we need to go for annealing treatment right here you see here this is the annealing treatment has been done for about an hour and what is shown here basically is tensile strength as a function of the you know tensile strength as a function with you know temporal annealing temperature 100 300 500 700 degree Celsius temperatures.

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And as you see tensile strength will slowly decrease in a cold work material tensile strength is very high something like about 550 MPa, but it slowly goes down and as the

temperature increases distant decreases slowly and reaches a flat regions at a very high temperature.

Subsequently, similarly or rather in opposite way not similarly ductility increases it is very low it is about 20 percent then it increases increase the reaches of flat regions which is about 50 percent of ductility. So, by you know by simply heating it up or annealing it any cold work material we can recover the ductility subsequent sufficiently. So, that material can be used or even if further deformation are required that can be imparted what happened any many of these deformation processes the deformations are done a stepwise manner. Basically idea is to if the material has low ductility deform by certain amount then you go for annealing treatment to increase the ductility again you deform. So, that is this treatment which is known as heat treatment or annealing treatment at higher temperatures is basically made or meant for recovering the ductility by doing something.

Now, our task means phase transformation our basic things is aspect for which were discussing basically understand what actually happens when you heat a material at higher temperature after the cold work or after the deformation at lower temperatures. So, normally things which happens in a material can be categorized into 3 subsequent 3 separate classes first one is known as recovery, second one recrystallization and third one is grain growth.

So, at lower temperatures when you annealed the cold work material the processes which happens inside the material is termed as a recovery we will discuss what is that in a minutes time and then if you hit further recrystallization happens. Now recrystallization is formation of the new grains basically nucleation and of the new grains from the deformed grain and subsequently if heated at a very high temperature these new grains grow very fast.

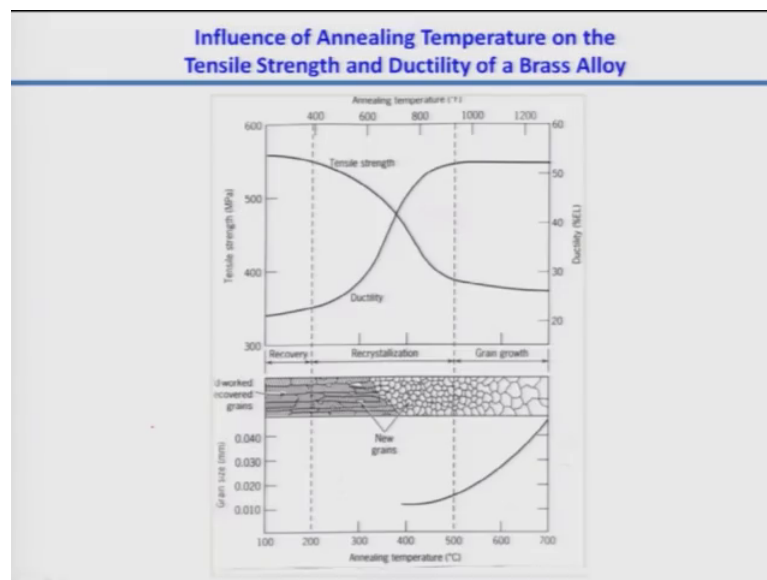
So, therefore, cold working of these materials leads to lot of defects and these defects actually drive the system or material for recovery recrystallization and grain growth kind of structures. As you say the during recovery what is actually happens written here recovery is basically a process in which physical properties like electrical conductivity corrosion resistance are recovered, but not the strength. As you see as tensile strength has not decreased much ductility has improved little bit from about 20 percent to 23 24

percentage, but strength has not change substantially what has change what will be changing is basically the physical properties like electrical conductivity corrosion resistance many other things.

So; that means, something happens to defect structures in the recovery stage which leads to improvement or change in the physical properties. So, what has actually happens as I already told you dislocation is the primary you know mode of deformation or primary means of deformation in the metallic materials. So, therefore, during recovery the dislocations actually moved little bit and find lower energy arrangements, atoms diffuse and point effects like you have industrials or vacancies their numbers will decrease or otherwise; that means, what that mean there is a rearrangements of the defect structures that is what happens in a recovery stage.

Let us discuss a little bit more what I am showing here is a for a situation of a brass, brass is nothing but a copper zinc alloy it gives basically alpha brass; that means, zinc concentration will be of the order of 40 percent maximum.

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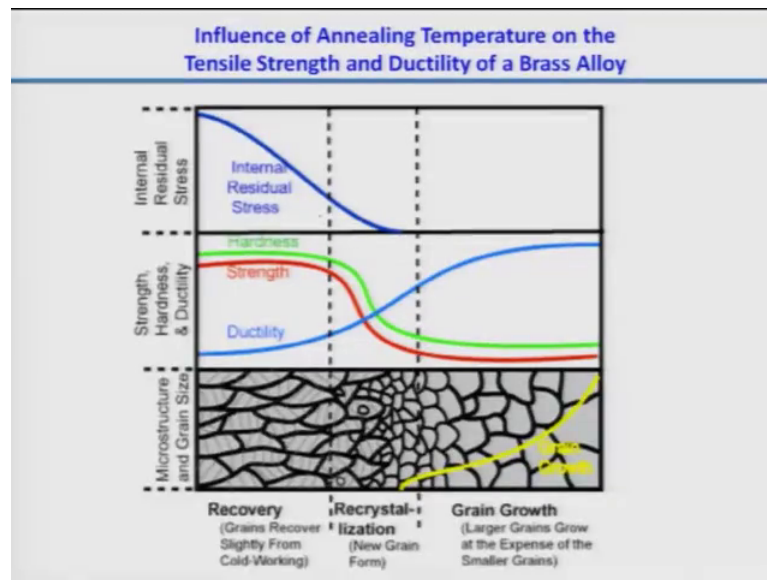
So, if you take a simple brass deform it and then subsequently anneal at temperature from suppose 300 to about 1200 degree Fahrenheit that is equivalent to about 700 800 degree Celsius temperatures. So, tensile strengths slowly decreases as you have seen earlier ductility improves.

So, what else I am showing in this view up is that the kind of grain structures and defect structures inside the material as you see here during recovery stage there is something some kind of a defects reorganization happens here. You can see here in this case defect reorganization happens that is means the defect structure which represent dislocations not things point defects they reorganize themselves and then once you heat it up to high temperature something like 300 to 400 degree Celsius temperatures new grains forms that is what is known as recrystallization and I am heated up to beyond 500 background screen scroll we will discuss each of these things separately. So, let us first discussed about the recovery what actually happens and recovery same thing is shown here. So, as you know any material if you deform material is a continuous things; that means, the grains are actually continuous they are attached to each others.

Now, you are putting a stress into the material. So, as the grains are continuous if you put a stress only way they can be remaining you know in touch with each other is by deforming them or generating some kind of stress inside and these stress which is generated because of these aspects known as internal stress. Internal stress is basically stored in terms of a deformation energy like dislocations point effects that is why it is stored. So, this energy which is stored in the material needs to be removed, the non material will regain its ductility plus ability are changes material properties

So, as you have seen the ductility and increase the increases as a function of temperature similarly the internal stress which is presence will also decrease, as you slowly heat it up.

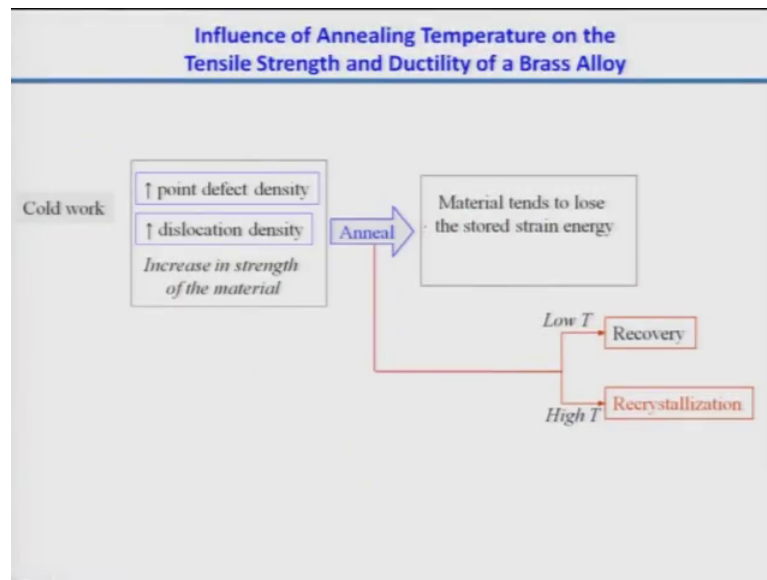
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So, as in the recovery stage internal cell undergoes a huge decrease something like 90 percent internal stress basically are removed during recovery stage. Simply by rearrangement of the defects and in the recrystallization stage the remaining part because you are forming a new grains remaining part is completely removed. As subsequently there will be no internal stress remain remember if there is a internal stress present inside the material deform in a material subsequently is very difficult why because internal stress will resist their further deformation. Not only that, because the internal stage resist is present inside the material if it is tensile in nature it can lead to cracking of the material failure of the material. So, that must be removed and this is done by simply heating up.

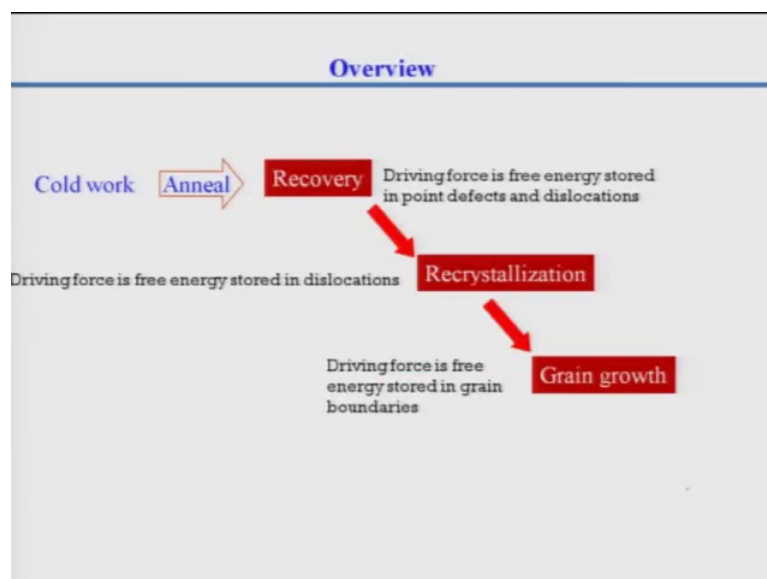
So, simple in the recovery stage what we shown the recover the internal stages extensively removed and that is done simply by simply by removing these 2 energy.

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So, in a nutshell what I can say the point whatever happened in a cold work stage the point if a density increases the dislocation density increases this will lead to increased strength if a annealed material tends to lose this energy and if a annealed lower temperature it is called recovery panel high temperature called recrystallization panel remain higher temperature much higher then it will lead to grain growth right. This is 3 stages you must remember.

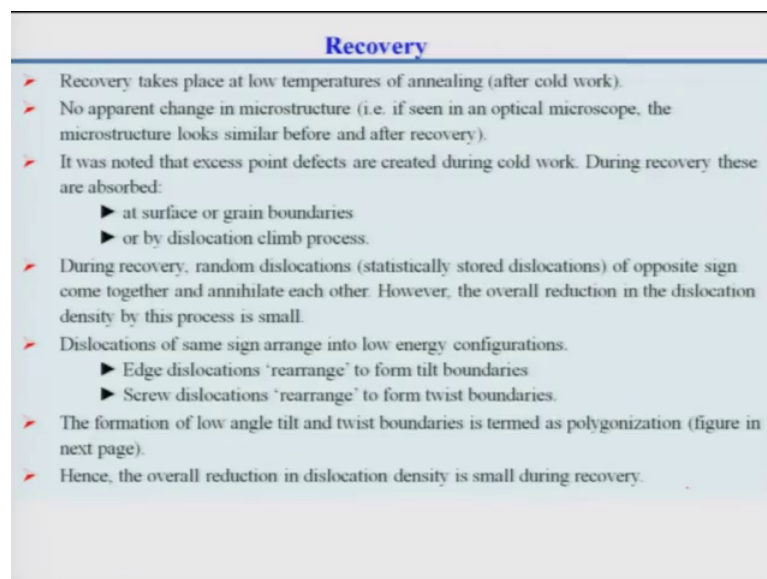
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So, the annealing stage driving force is basically what the free energy stored in the point defects and dislocations that is the driving force for recovery because cold working lead to lot of this defects structure. And then the recrystallization basically is happens because of the driving free energy stored in dislocations and the grain growth really happens because of the free energy stored in grain boundaries because your line about grain boundaries because of the small size grain forms the recrystallizations and higher temperature they want to remove that can be a grain boundaries. So, therefore, grain starts growing.

So, as per driving force is concerned for the fare fastest ah aspect recovery crystallization is the cold work energy which is stored in the material irresponsible for the transformations. But in the last stage grain growth stages basically energy stored in the grain boundaries is basically used as a means for the phase transformations.

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Recovery

- Recovery takes place at low temperatures of annealing (after cold work).
- No apparent change in microstructure (i.e. if seen in an optical microscope, the microstructure looks similar before and after recovery).
- It was noted that excess point defects are created during cold work. During recovery these are absorbed:
 - ▶ at surface or grain boundaries
 - ▶ or by dislocation climb process.
- During recovery, random dislocations (statistically stored dislocations) of opposite sign come together and annihilate each other. However, the overall reduction in the dislocation density by this process is small.
- Dislocations of same sign arrange into low energy configurations.
 - ▶ Edge dislocations 'rearrange' to form tilt boundaries
 - ▶ Screw dislocations 'rearrange' to form twist boundaries.
- The formation of low angle tilt and twist boundaries is termed as polygonization (figure in next page).
- Hence, the overall reduction in dislocation density is small during recovery.

So, recovery takes place at lower temperatures from handling as you have seen just subtract. In fact, in recovery these are things happens first thing is that there will be no apparent change of micro structures; that means, there will be no change in the phases, no change in the grains size, no change in the boundaries other things. That means, if you have seen in a microscope you will not see any change in the microstructure because micro structure observed in the microscope.

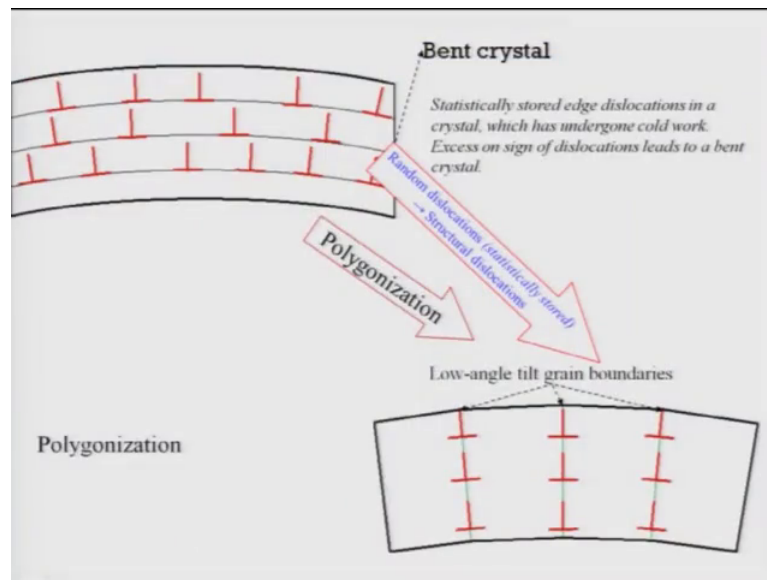
But what will happen is that the excess point defects which are created during these plastic deformation or cold working or warm working processes will be absorbed at the grain boundaries or at the surfaces or even some of the defects like dislocations can also be removed by climbing processes. Dislocation can actually move by you know gliding on a slip plane or they can climb because of diffusional processes as you have probably studied in a mechanical metallurgy. But forget if we do not understand anything this point defects actually point defects or even the dislocations which are line defects they can actually get absorbed on the surface grain boundaries.

Till recovery random dislocations just such statistically stored number of dislocations will be there which are opposite signs they can come and anneal out; obviously, annihilate. Dislocations which are this kind of sign or this kind of sign they can come close and they can anneal out very simple this one at the top is the one which is shown like this dislocation that this one has an extra row of atoms coming from top to bottom the one this one has one extra row of atom kind for bottom, when they come together both the extra row atoms match and the dislocation is annihilated.

But overall dislocations reduction in dislocation density is very very small although they get removed dislocation same sign can arrange important aspect dislocation of the same sign that is for the opposite sign they can come annihilate. Same sign can arrange they are rearrange themselves to form low angle grain boundaries screw dislocations tilt one edge once the screw dislocation can arrange themselves to form a twist boundaries. So, the formation of these low angle grain boundaries or twist boundaries this is known as what is known as a polygonization in the literature. I am showing that next picture in the same things. So, this will lead to a gradual reduction in these (Refer Time: 26:01).

So, what is polygonization? Let us look at it in a very brief way you know that in a material in a basically deformed material they have a lot of and that is plastic deformation energy or strain energy stored by in terms of dislocations these are all dislocation of same type edge dislocation of same type.

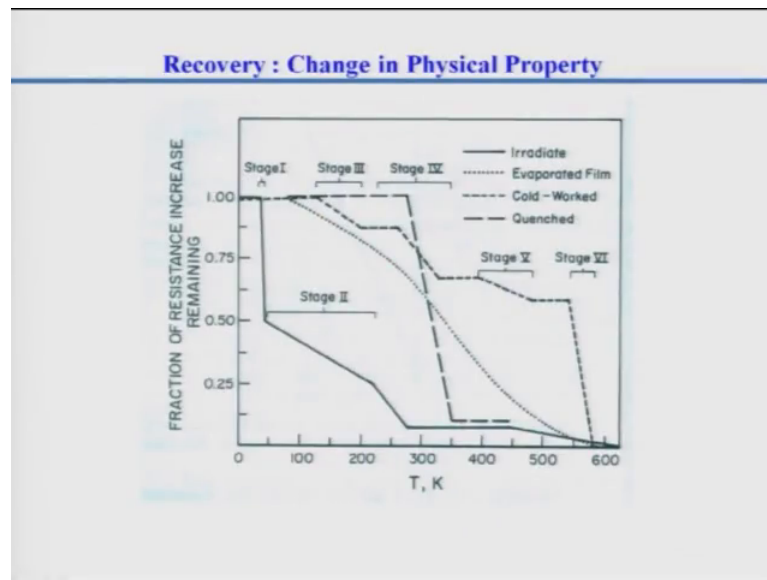
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Now, statistically stored edge dislocations in a crystal can actually lead to you know huge amount of energy stored and this is what a reason for the decrease in ductility. Now, what can do this dislocation this dislocation can actually easily arrange on top of each other instead of arranging on a line you see there are arranged on 3 different lines they arranged on vertically, these things are coming vertically top of each other.

So, as they arrange topically about vertically top of each other and, therefore, they form a low angle teal boundaries very simply we could go back my own textures in this course about the boundaries steel boundaries twist boundaries till boundary is nothing, but a vertical arrangements of the dislocations. So, this is the way these dislocations can actually arrange nicely and this is known as polygonizations. This has been observed in the actual literature actual micro structures. So, polygonization means to decrease the store energy as you see here. We have not decreased the number of dislocations or density dislocations by this process what is happening here is basically rearrangement of a dislocations in a certain specific manner, so that say energy of stored in this part of the crystal is removed energy is basically rearranged along in terms of the dislocations lining along the vertical line, so obviously, this leads to change in the physical properties.

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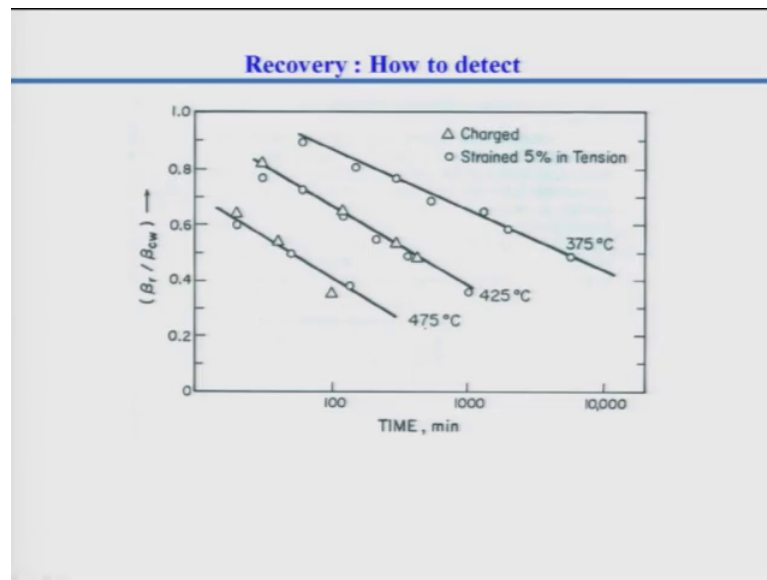


One of the physical property which is important is the resistance or conductivity inverse resistances estimate is not a conductivity. As you see here let us look at a cold work material this one which is given by this line. So, there are several stages of these change in the cold this resistance initially resistance will not change much if you heat up to room temperature. But as you go beyond room temperature there is a sharp decrease this is for anneal actually there is a sharp decrease of the resistance plastic decrease because that is; obviously, initially there are lot of defects so a stability was very high as the defects case rearranged this stability decreases slowly.

So, and similarly for the other samples like if you look at if you look at basically you can actually have a other stages you see here there are stage 1, stage 2, stage 3, stage 4, stage 5, stage 6, but here they are actually 2 to 3 stages 1 2 3. So, depending on the type of materials how the dislocations get arranged how these point defects get arranged there will be different number of stages that is possible. But important aspect is that physical parameters do gets change not the microstructural parameters that is what you must understand.

Now, how we can actually do that measure it we can measure it using X-ray diffraction basically.

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This is at annealed sample at different temperature (Refer Time: 29:29) 75 to 475 degree Celsius temperature different time scale up to about 10,000 minutes. What is plotted here is basically the you know for the ratio of the FWHM or pulled with a maxima for the cold work for the 2 materials - one is annealed another is cold work. This is a ratio is plotted. As you see the ratio is slowly decreasing as you go along the timescale.

So that means what; that means, that the strain increase the material inside the material is slowly decreasing because the pulled with have maxima and of the peak in X-ray diffractions it tells you the when the grain size is not changing much it tells you the strain induced in a material. So, this is the way one can actually calculate the strain reduction the strain in a material.

So, let me stop here, but let me just tell you that we have simply discussing or whatever do we discussed today is that cold working is in evident in the material processing. Many of the materials are cold work because we need to give steps and this cold work leads to lot of defects generations of the material, and effects will lead to decrease the ductility and many other property like toughness.

So, in order to regain that we need to annihilate at higher temperatures during annealing you can have many kinds of as you know possibilities like you can have reorganize of the defect structure which can lead to recovery, you can also generate new grains inside

the material which is known as recrystallizations or because of these grains are very small they can grow at higher temperatures what is in a can happen is known as grain growth.

Recover is the first step in these annealing techniques, recovery leads to reorganization of the defect structure without much change of the microstructure and this leads to change in the physical properties like electrical conductivity resistivity or corrosion resistance. So, we will discuss more about recovery little bit more and further we will discuss about crystallization in subsequent lectures.