

# **Nanostructures and Nanomaterials: Characterization and Properties**

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## **Lecture - 5**

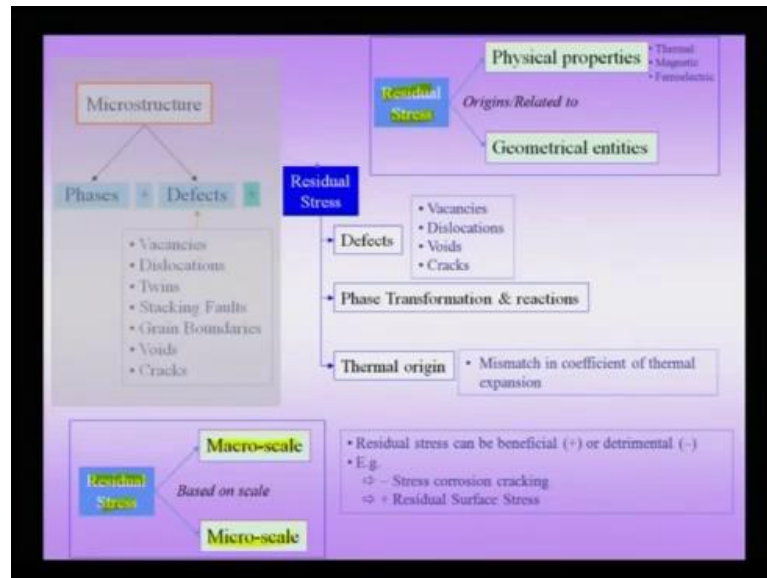
### **Introduction to Nanomaterials (C1)**

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- **Overview of fundamental concepts (continued)**
- **Importance of residual stress on properties**
- **Change in properties across lengthscales: polycrystalline Cu, Demagnetized Fe sample**
- **Traversing across lengthscales (Going from an atom to a component): Fe atom to a gear wheel**

Let us now proceed to the next important component of the micro structure, which is residual stress and its distribution. We will see how residual stress deserves a place equivalent to facets and its distribution when it comes to description of a micro structure and how it can influence the properties of a material, the origin of residual stresses.

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If you look at the slide at the top, right hand side can be arising from various physical properties like thermal origin, the magnetic origin could be ferroelectric ferromagnetic etcetera. It could also arise from geometrical entities example of some of which we have already seen and may be some more we will consider very soon. Traditionally, residual stress has been classified based on the scale at which it exists, typically it has been classified as the macro scale and the micro scale. So, let us start with what is meant by residual stress before we go on to consider the classification of residual stresses.

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**Importance of residual stress**

- Residual stresses are those which arise in a body in the absence of external loads or constraints.
- Processing will influence all the three origins of residual stress.
- These origins also represent various lengthscales.

Vacancies and dislocations give rise to stress fields at the atomic scale, while residual thermal stresses could pervade the entire component.

Some points in this regard are:

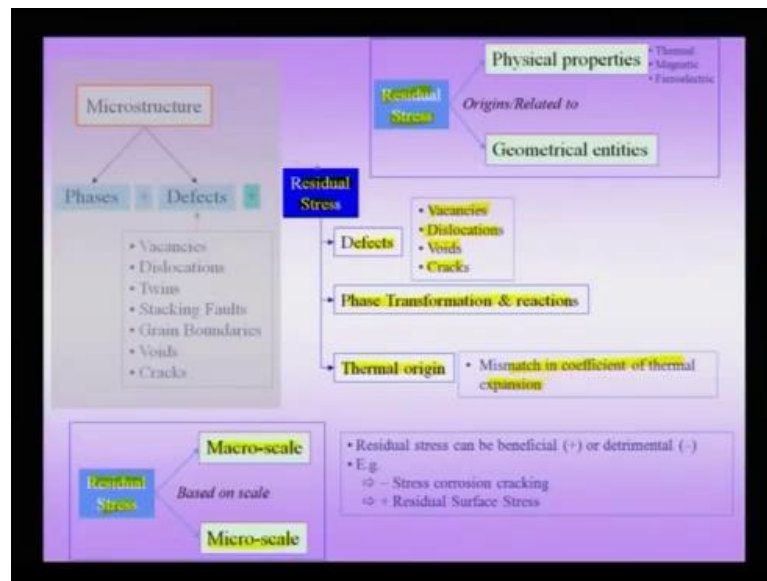
- the stress fields associated with GP zones in Al-Cu alloys is of the same scale as dislocation stress fields
- large cracks in the material can lead to macroscopic stress fields, while microcracks may have much small effective region of stress fields
- in micron sized components, the scale of thermal residual stress may be expected to be smaller than that in their large scale counterparts.

• In the example of GP zones in Al-Cu alloys, the stress/strain fields are intricately associated with the distribution of GP zones (- the phases); which further highlights the importance of adopting a definition of microstructure as done in here.

*[Note: Cracks themselves are not sources of stresses; they merely amplify a far field stress. As they can amplify a small far field stress (which could be residual in nature), they have been included in this section].*

Residual stresses are those which arise in a body in the absence of external loads or constraints that is if I of course, put a load on a body or I give a displacement to a body. I do expect residual stresses to come about, but the stresses which remain after all the external constrains and loads have been removed s called residual stresses. Hence, residual stresses are those which arise in a body in the absence of external loads or constraints.

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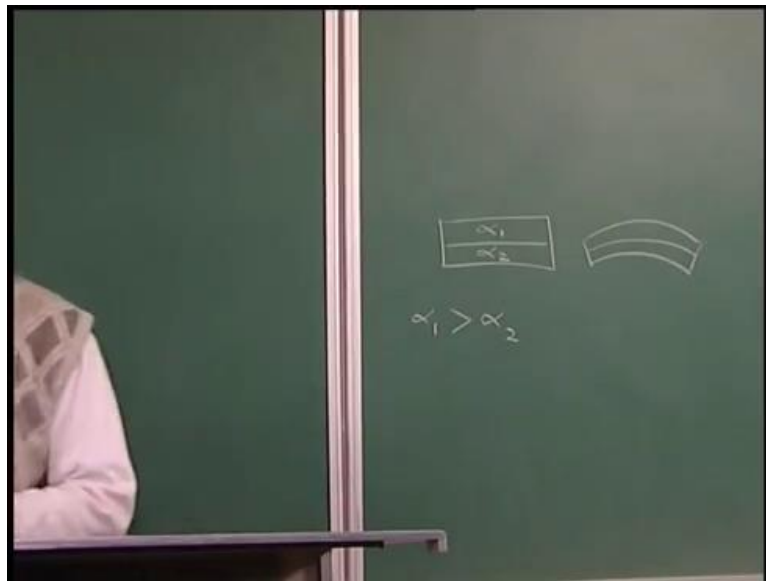


Now, why is that we need to classify residual stresses, we need to understand that there are multiple origins of residual stresses some of which as we may say can be at a very small nanometer length scales. Some of them can be at the length scale of the entire component; some of them are related to the process. These are occurring in the material like phase transformations and reactions, while others of them are related to the differential thermal cooling which the sample feeds.

So, residual stresses can be thought of as those arising from defects and these defects include dislocations vacancies interstitials etcetera. We have included voids and cracks in this classification and we will soon see that what you might say is a slight difference in the way voids and cracks give rise to residual stresses as compared to dislocations and vacancies. We will talk about that very soon phase transformations and reactions can also give rise to residual stresses and we will see that how this kind of a residual stress is important from the perspective of the micro structure and the micro structure evolution.

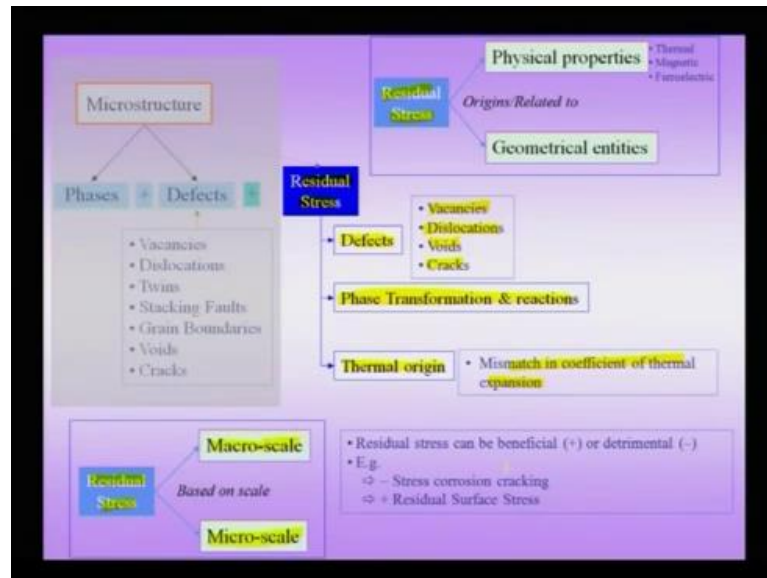
The usual origin of residual stress which we often talk about when we talk about warpage of components etcetera is what is we call the thermal origin, which comes from the mismatch in coefficient of thermal expansion. We have a bi metallic strip and when we heat this strip the coefficient of expansion on either side of this bi metallic strip will be different, therefore the strip can get into bent configuration along with the presence of residual stresses.

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So, I have suppose a bi metallic strip and the coefficient of expansion of one these alpha 1 and the other one is alpha 1 and alpha 1 can be greater than alpha 2. Now, suppose this strip is heated, then this will tend to get into bent configuration and even after bending the complete stresses may not be relieved. Especially, if alpha 1 and alpha 2 are not in this plane or geometry, but in certain more complicated geometry and therefore you may have residual stresses coming purely from what you might call mismatch in coefficient of thermal expansion.

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An important point to be kept in view when we are talking about residual stresses is a fact that often in an engineering sense, it is conceived or it is perceived that residual stresses are bad for the body. It can cause warp age of the component, it can cause failure by propagation of cracks etcetera, but we should clearly bear in mind that residual stresses can be beneficial or can also be detrimental. So, both these possibilities exists and we will explore this in a little bit of detail in this coming slides.

So, let me summarize this slide number one residual stresses are important and they have to be considered within functional framework of definition of a micro structure number. Two residual stresses can be classified based on their occurrence on scale, which can be macro scale or micro scale and all the other length scales which pervade between the scale of the vacancy or the atom to the scale of the entire component.

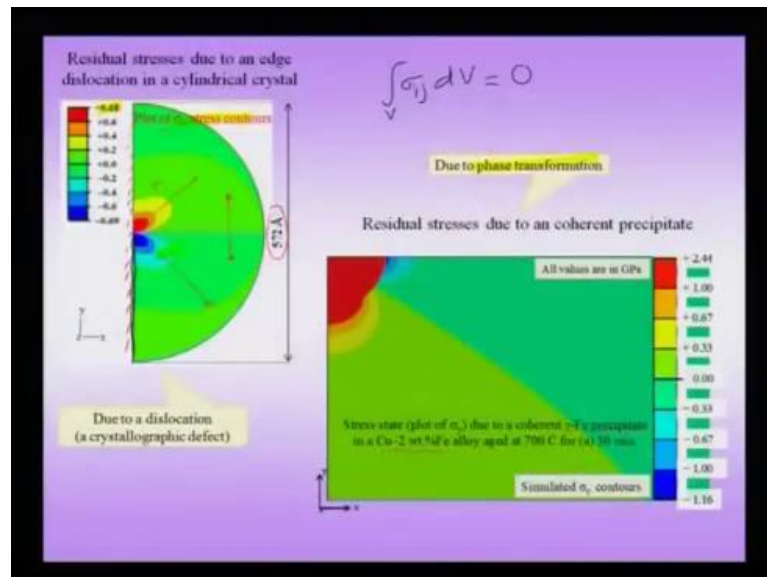
They can arise because of various physical properties like on during magnetic phase and you have switching on of a magnetic field, they can arise from geometrical entities. When I mean geometric entities, I mean precipitates etcetera and all these origins have to be kept in mind because the kind of origin will tell us under what circumstances this residual stress is arising and how if I want can I engineer this residual stress.

Some sort of a classification of these residual stresses can be based on those arising from defects in the material like vacancies dislocations etcetera, those which arise from phase transformations and reactions and phase transformation. I mean it could include austenite

two martensite phase transformation precipitation reaction or many of the other possible reactions including oxidation. They could be of thermal origin, which basically means that you have a mismatch in coefficient of thermal expansion.

The most important point with regard to residual stress is that they could be extremely detrimental for the body as we shall see using examples, but additionally they could also be beneficial for the body. So, whenever I have beneficial effect of residual stress, I can actually engineer the residual stress so that properties of my component or my material improve, now there are two examples given here.

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One is that arising from dislocation, which is shown in the circular body and semi circular body in the left hand side, and the other one is that arising during due to a phase transformation. If a dislocation is present in a material, then there are compressive and tensile stress fields, which are present associated with the dislocation and typically these would on the whole cancel out.

In other words, if I perform an integral like integral of  $\sigma_{ij} dV$ , then this integral over the entire volume will give rise to a 0 net residual stresses. In other words, if there is a free standing body and I have regions of compressive stresses, then there has to be tensile stresses, somewhere else in the body so that the overall integral of  $\sigma_{ij} dV$  turns out to be 0 in the case of dislocation. You can see that the extra half plane, suppose

it is present, I am talking about here, which means that the extra half plane could be present on the bottom side.

Therefore, there are compressive residual stresses in the region of extra half plane and there are tensile residual stresses in the region other side of the half plane. Now, this is of course, an edge dislocation and the stress we are plotting is here is the  $\sigma_s$  stress close to the core of the dislocation. You can see that the stresses are very high in the magnitude, they can go up to the order of 6.8 Giga Pascal or more. On the other hand, if I go far away from the dislocation core, I see that the stress is decreased.

So, if I go away from dislocation core, then the stresses decrease and after sometime I can think of as these the effective region of the dislocation dived on and I do not have to anymore talk about the stress field of a dislocation. This stress field plotted here is that for a dislocation in a finite cylindrical body and of course half the cylinder or half the circle is shown here for convenience. The other half is exactly symmetrical to this half, so this is mirrored here with respect to the stress fields.

In an infinite body, the entire half space for instance the top half space would be completely tensile and the bottom space would be completely compressive, but in an finite body, you will notice that here there is tensile stresses. This means there is positive value, but farther away from the core of the dislocation, actually it becomes compressive. So, this is one nice example which is the stress of the dislocation, which is present in the absence of any external loading.

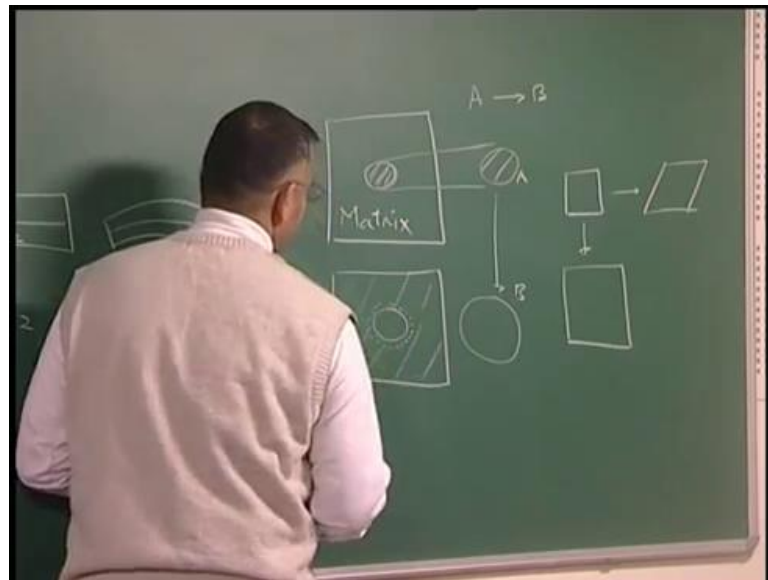
Of course, if I apply an external sheer stress, this dislocation may be driven because of the interaction of the externally applied sheer with this dislocation stress field and the figure on the right talks about the stresses coming due to phase transformations. Now, of course, depending on the kind dislocation we are considering or it could be a mixed dislocation the stress field, which we are seeing vary considerably, but here we have considered an edge dislocation in a cylindrical body for convenience.

If you notice that the size of the body is very constraint, it is actually of about 50 or 60 nanometers. Now, this defect this stress field we are seeing is because of a crystallographic defect, which is a dislocation. On the other hand, the other example we are considering here is due to a phase transformation. That means that there is originally

a barren phase in which there is a small region of material, which is transforming to a different phase.

For instance, in this example we have shown this stress plot for coherent gamma precipitate forming from an copper aluminum alloy. So, you have copper aluminum alloy from which nearly pure gamma precipitate comes out and the stress plot is showing that this gamma stress precipitate is associated with. You might call this a coherence stress and therefore, the body is stressed, so let us consider briefly what the origin of these stresses in the board is.

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So, we have a body in which I can think of a small region, which is transforming of course it need not be a spherical region, but for convenience we will consider a spherical region. Now, suppose this body this volume whatever I marked in shaded is actually going to transform from one phase to another, for simplicity I can think of it as going for instance from phase A to phase B.

There is no reason to believe that phase A and phase B have same volume, therefore I can make a what is known as shell B cut, I can take out this region of material. So, this a hole in a medium, now I allow this region of material to transform and the transform volume could be larger smaller. Of course, in general it could be associated with both sheer and volumetric strain, so the new body could have new shape. Also, it could have a



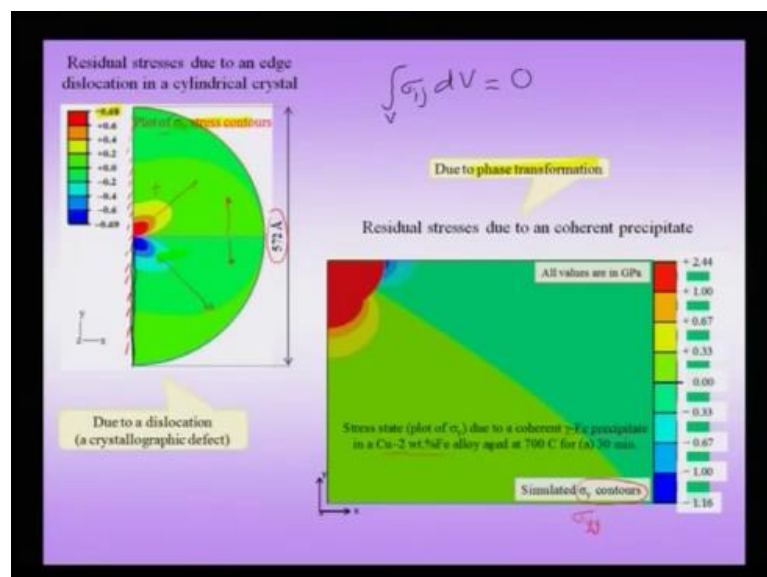
new volume bigger volume or a combination of both in other word sheer and both dilatational may be involved with this phase transformation.

For now, just for the simplicity I will consider only the dilatational strains, so the body which was originally occupying the volume  $v$  here could be occupying a larger volume or a smaller volume. Therefore, after a phase transformation say for now it occupies a larger volume, so this is now phase at which has got transformed into phase B.? This is now a free standing body which is now allowed to freely expand, but in reality this is actually embedded in this other material, which is now my matrix material.

In other words, this volume B, this material B has to again fir into this hole from which it arose, originally this implies that the surrounding matrix will actually try to compress down this volume because this tries to occupy a larger volume. As the example, I have considered and the material we want to expand outward and this will come to an equilibrium configuration wherein a certain. For instance I will just draw by dotted line the equilibrium configuration this leads to a residual stress state in a material which is what I am showing you as they plot.

So, let us return to the slides and see how a stress plot looks if such a material were produced by a phase transformation. So, this is what we call a coherent precipitate wherein there is a continuous lattice plane matching between the precipitate and the matrix.

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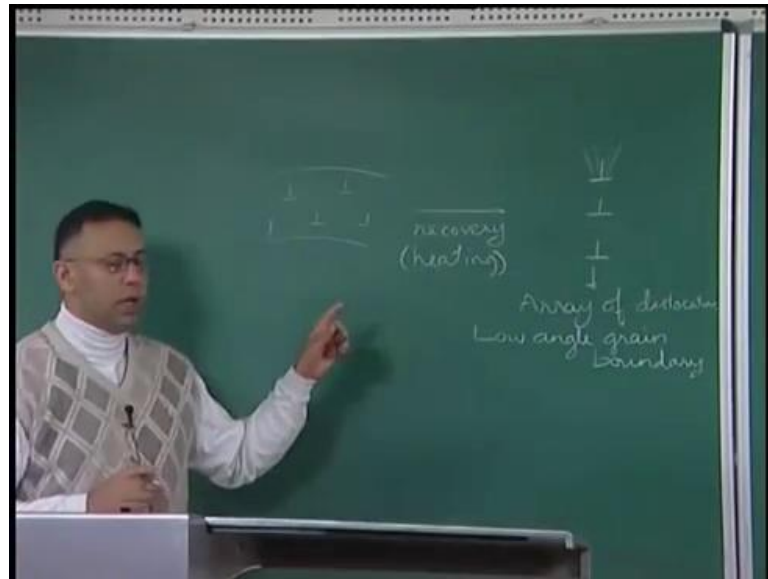
Now, you can see that the region where the precipitate sits has been stressed in a tensile fashion which is shown by the red color contours, but importantly also that the matrix has also been stressed. Here, we are plotting actually the  $\sigma_y$  contours here or which you want to fully write is  $\sigma_y$  contours and you see that regions of the matrix have been stressed in a tensile fashion. There are other regions in the matrix, which are in compressive stresses, but the important thing to note again is that in the entire body there is stress failed.

This means not only the precipitate is stressed, but also the matrix is stressed and this effect of the stress field of the precipitate can have considerable influence the way the precipitate interacts with other defects in the material. This could be also edge dislocation sitting in the matrix and in fact it could actually attract dislocation sitting in the matrix and it may on further growth go from a semi coherent to a semi coherent state. This edge dislocation from a matrix or a dislocation loop from a matrix could actually play the role of an inter phrasal misfit dislocation.

So, we are clearly seeing that there are crystal graphic defects like dislocation which have residual stresses and unless I remove a dislocation from the material, these the dislocation will continue to have these residual stresses.

We saw that the entities like precipitates can have residual stresses of other origin and therefore, giving rise to certain interaction of defect and these are long range stress field interactions of defects. A nice example perhaps which we have about which we have talked earlier was the fact that how dislocations which are present in the matrix in the random fashion in the same sign. The dislocation of the same sign present randomly in the matrix can actually attract each other and form a low angle grain boundary, when you actually heat the material and this process is called recovery.

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So, I will just draw the figure once again to remind you that how suppose I had a bent crystal in which there are dislocation sitting randomly in the crystal. So, if you do a recovery heating process, then these could come together and arrange in a form of a low angle grain boundary. So, there will be a slight disorientation in the angle between the two sides of the plane and this is now the array of dislocation forming a low angle grain boundary.

So, we had considered this example before, therefore this is possible through the of course, through the thermal activation, but also equally importantly by the interaction or the long range stress fields of the dislocations. Now, we go to certain important points regarding what is the origin of these residual stresses and the important question that is why I am including the residual stresses and its distribution as a part of the definition of a micro structure.

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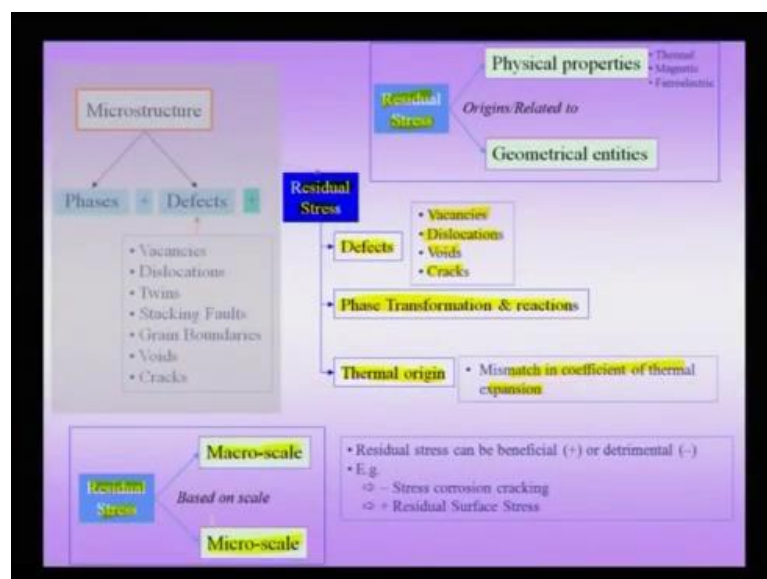
- the stress fields associated with GP zones in Al-Cu alloys is of the same scale as dislocation stress fields
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- in micron sized components, the scale of thermal residual stress may be expected to be smaller than that in their large scale counterparts.

- In the example of GP zones in Al-Cu alloys, the stress/strain fields are intricately associated with the distribution of GP zones (~ the phases); which further highlights the importance of adopting a definition of microstructure as done in here.

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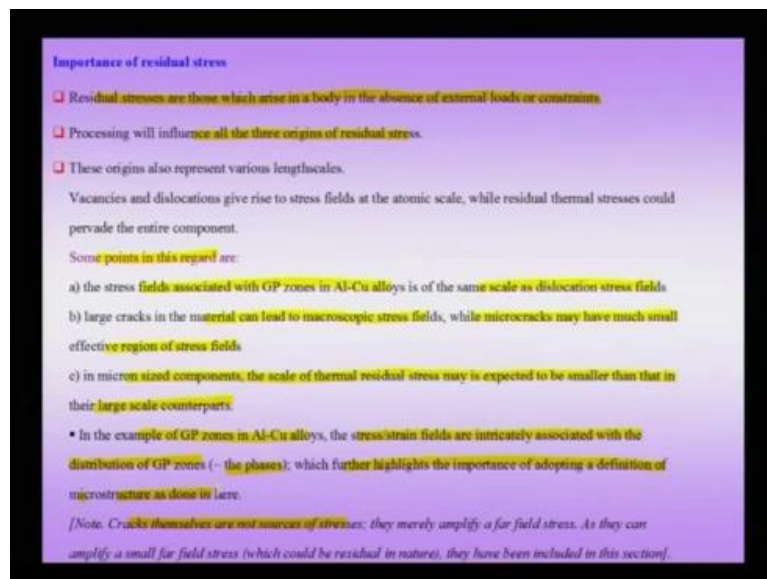
We have to note that all three forms or all three origins of residual stresses, when I am talking about three origins I am talking about those arising from defects those arising from phase transformations and those having a thermal origin. Therefore, when I am making doing a thermo mechanical treatment, which can be rolling forging hot rolling etcetera which is giving rise to my final product or a component, this processing will actually affect the all three origins of the residual stresses. So, this has to be kept in mind additionally we also have to keep in mind that these origins also represent multiple length scales we declassified earlier.

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We had noted that residual stresses can come from the entities at the micro scale or entity at the macro scale, but these are what you call very crude classifications and we noted that across all length scales, you could have residual stresses. The length scales we are talking about is right from that at which a vacancy exist to the scale of precipitates and dislocations to the scale of the entire component. So, we have to remember that therefore, residual stresses can pervade across the entire gamut of length scale available to the material.

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So, we have to note some important points in this regard the stress fields of associated with GP zones in aluminum copper alloy, for example is the same length scale as a dislocation stress fields. Now, GP zones as we know are copper rich zones in an aluminum copper alloy, which comes about when you actually age your super saturated solid solution. So, these are copper rich zones sitting on specific kind of crystallographic planes, but since copper rich zones have a different lattice parameter as compared to matrix aluminum copper alloy, which is basically a aluminum rich alloy.

Therefore, there are going to be residual stresses is allowed, but this scale of residual stresses or the effective length scale at which these residual stresses reside is of the same order of the length scale as a dislocation stress fields. Now, if it had large cracks in a material this can give rise to macroscopic stress fields while micrographs may have a smaller effective region of stress fields.

Therefore, we have to note that an important point with regard to cracks and voids being associated with residual stresses is that actually cracks and voids are themselves not source of residual stresses cracks and voids. They are nothing but stress concentrators or stress amplifiers in other words, if I apply a far field mean stress then a crack present in a material will actually amplify these stresses.

As we know if there is any other origin of residual stress in the material, for instance we saw the precipitate and if the cracks present in the vicinity of this precipitate this crack will may tend to amplify the presence present residual stresses. In other words, though cracks and voids themselves are not associated with residual stresses since a small amount of mean field stress can actually be amplified by crack. We need to talk about we may want to include cracks and voids another kind of what you might call pores in the material into the definition or into the origins of residual stresses.

So, this is very important, so once again cracks and voids are themselves not associated with residual stresses, but they actually tend to amplify far field or mean field stresses. We have to we tend to include them in the definition of residual stresses or the origin of residual stress in micron sized components, the scale of thermal residual stress may be expected to be small than that in their large scale counterparts. So, as we know now that because of nanotechnology, we have components which are actually in a nanoscale, we have components in the micro scale.

Of course, we have our usual regular components and devices which function is the macro scale like we could be talking about the gas turbine engine or the blades of a glass turbine engine. These are of course, in the large meter length scale, but there could be components which are called for instance the micro electro mechanical system, which exists at much smaller length scales. Therefore, here residual stresses the length scale of the residual is much smaller, but that does not mean that the magnitude of the effect of residual stresses could be neglected in these.

In fact, if the length scale is smaller sometimes the effect is actually more amplified and we may actually want to take into account the residual stress in a more rigorous fashion in the example of GP zones and aluminum copper alloys. The stress strain field are intricately associated with the distribution of GP zone that is the faces which further

highlights the importance of adopting a definition of micro structure, wherein we are not only talking about the phases and their distribution.

The defect structure and their distribution, but also the residual stress and distribution, so to repeat the last point in aluminum copper alloy, wherever there is a formation of GP zones or the theta precipitate or the theta precipitate. The distribution of phases is very intricately associated with the distribution of the residual stresses.

This is coming from the phase transformation and therefore, in some sense the distribution of residual stress is closely associated with the distribution of the phases themselves. Therefore, this definition of residual stress, which we have or the definition of micro structure which we have adopted makes a lot of sense.

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The reason for elevating residual stress to be an integral part of the definition of microstructure will become further clear in the next two examples considered.

- It is well known that nucleation is preferred at any of the high-energy sites in a material (heterogeneous nucleation). Typical examples of heterogeneous nucleation sites are: surfaces, internal interfaces (grain boundaries, interphase boundaries, stacking faults etc.), crystallographic defects (dislocations), cracks, voids etc. Stressed (and hence strained) regions also can act like heterogeneous nucleation sites.
- In corrosion formation of a galvanic cell leads to corrosion of the anode. At the level of the individual phases, one phase may be more anodic as compared to another, thus forming a 'micro'-galvanic cell. Similarly, tensile regions in the sample may behave anodically with respect to compressive regions in the sample which can behave cathodically.

The figure contains two 3D block diagrams of a sample. The top diagram shows a flat surface with an 'Anodic phase' on the left and a 'Cathodic phase' on the right. Below the surface, the reactions are given as 'At anode:  $M \rightarrow M^+ + e^-$ ' and 'At Cathode:  $M^+ + e^- \rightarrow M$ '. The bottom diagram shows a similar setup but with a red wedge under the anodic phase labeled '(tensile)' and a green wedge under the cathodic phase labeled '(compressive)'. The reactions are the same as in the top diagram.

We will consider a few more examples to see why we need to elevate residual stress to the level or to be an integral part of the definition of a micro structure. In these two examples, one I consider the growth and nucleation and growth of epitaxial islands and the second, I consider is the presence of precipitate phases in a material. In the growth of epitaxial islands, so what happens is that you have an epi substrate on which there is a small island, which is nucleating which grows into the form of these mounts. So, let me draw that using a figure here.

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So, typically there is a substrate and on this substrate I deposit some material and I could be using a technique like molecular beam epi taxi and this is deposited material has three modes of growth possible. Either, it can grow in the form of a layer by layer grow or it can grow first as a layer followed by formation of island on top of the layer or the third possibility, which we have shown here is a direct nucleation of an island and the growth of an island. Now, initially a small island, I will focus on one island a small island may be nucleated and later on with the progress of more and more additional more and more material this epitaxial island will grow.

When I am talking about epitaxial island, I mean that the substrate and the island have a very similar lattice parameter, typically which means there is a continuity of lattice planes from the substrate to the epitaxial island. In many of this process, what is done is that we do not have only one layer of islands after growing a layer of islands shown in the bottom most figures here.



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The reason for **elevating residual stress** to be an integral part of the **definition of microstructure** will become further clear in the next two examples considered.

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- In **corrosion formation of a galvanic cell leads to corrosion of the anode**. At the level of the individual phases, **one phase may be more anodic as compared to another, thus forming a 'micro'-galvanic cell**. Similarly, tensile regions in the sample may behave anodically with respect to compressive regions in the sample which can behave cathodically.

So, let me consider this figure one for instance, so here we have epitaxial islands which we have grown and after growing these epitaxial islands, we may go and put actually what is known as a capping layer on these islands. So, in other words you have a substrate you have an epitaxial island and a capping layer which completely buries my islands into the capping layer. In other words, person looking from top will not be able to see these buried island layers or the layers containing these buried islands. Now, if I go further and actually try to grow at a second layer, that means now I continue my MBE process.

After I have put capping layer, I can do my MBE process and try to deposit a second layer of islands in, of course what would one expect that these islands can randomly nucleate at some other position and start growing. Usually, it is seen that that does not take place and there are preferred directions along, which there is an alignment and that occurs because of easy direction of strain propagation. Now, I consider that this is my easy direction of strain propagation and therefore, what happens is that the next layer of islands nucleates exactly of top of the first layer. So, what is responsible for this alignment of this second layer of islands on top of the first layer this is the presence of these epitaxial residual stresses.

So, let me go through the process, again I have a substrate on which I grow epitaxial certain islands of course,, islands grow or we will continuously dependent upon other

issues which we are not considering here, but once an island has grown on one layer. If I put the capping layer and then I go on to grow a second layer of islands, I notice that the second layer will align itself in a particular fashion with respect to the bottom layer. Now, I consider it right above the buried layer, in other words the second layer is not randomly growing above the first layer; it is growing along some preferred directions.

That means there is something which is preferentially nucleating this second layer of islands, what is that it is actually the straining or the strain coming from the residual stresses which is the coherency stresses or the epitaxial stresses. This implies suppose I am talking about nucleation, we know that from our classical understanding of nucleation that nucleation is preferred at any of the high energy sites. In a material, this could be a grain boundary or this could be a dislocation or this could be a pre existing precipitate and all these forms of nucleation are called heterogeneous form of nucleation and surfaces.

As I cited surfaces, internal interfaces like grain boundaries interphase boundaries stacking faults etcetera are preferred sites and these preferred sites could also include crystallographic defects like dislocations and other kind of defects. We just talked about like cracks and voids from example of heterogeneous nucleation, we can see that strained regions in a material can also act like heterogeneous nucleation sites. That implies that they have a role, which is very similar to that of a defect or of a phase present in a material.

We see that we could have a second phase or interphase of a second phase, which could act like a nucleating phase or a dislocation could act like a heterogeneous nucleating site. Here, strainor which is originating from the epitaxial stresses is the origin of what you may call heterogeneous nucleation. Therefore, I would want to consider stress or residual stresses part of the definition of the micro structure like defects and phases and phase distribution.

Now, another example this is coming from what you may call corrosion and the formation galvanic cell in corrosion the formation of a galvanic cell lead to the corrosion of a nanoscale. That means the nano, preferentially dissolves when you actually have an electro chemical cell or a galvanic cell forming at the level of the individual phases one or more phases may be anodic with respect to the other. Thus they form a micro galvanic

cell, so when i talking about the galvanic cell, I could actually have a macroscopic electrolyte system, wherein there is one electrode which performs the role of anode another, electrode. This performs the role of a cathode, but this is what you might call a macroscopic galvanic cell, but a galvanic cell could also form at the micro structural level of the individual phases.

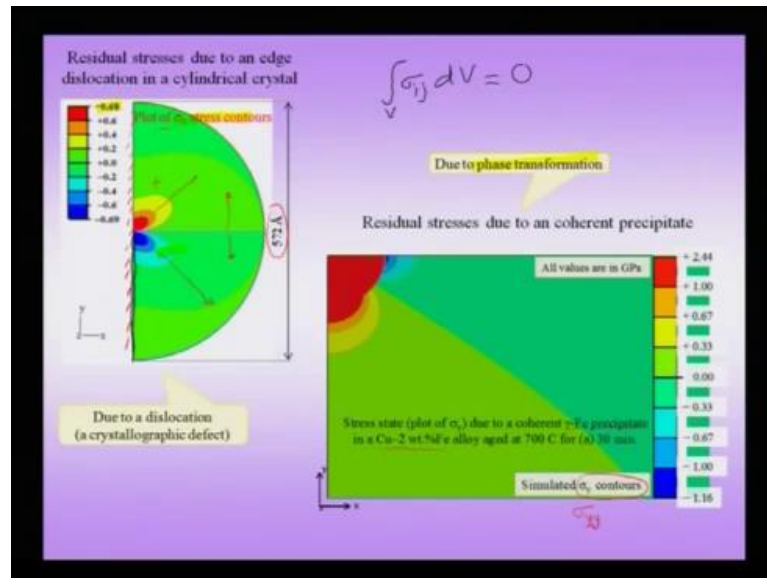
For instance, in the diagram shown in the right, you can see that there are two phases, one is being shaded dark. Now, I can call a phase A and there is fade, which is a lighter shading which is phase B, I assume that this is present in a blue electrolytic material as shown here. Now, there is no macroscopic galvanic cell here, we have an electrolyte medium and there are two phases. For now, I will assume that the phase a behaves is an more anodic as compared to phase B and therefore, in the presence of an electrolyte.

It will actually give rise to an electron, this electron will of course, combine with the metal in the electrolyte and deposit at the cathode. So, this will be the cathodic reaction and the anodic reaction is that of dissolution, so where is this electro chemical or galvanic cell forming this galvanic cell forming at the micro structural level. It is forming between two phases in the micro structure the phase A and the phase B and in the of course, in the presence of an electrolyte. So, it is very clear that when I am talking about corrosion this kind of a corrosion could actually be much more detrimental because now this is occurring at the microscopic scale because phase A.

For instance, this could be of the order of say 10 microns, it is not as something or 10 micron, which is not visible to the naked eye and since the corrosion is extremely localized. It could actually lead to a stress concentrator or could actually lead to a local damage, which is not directed and which could lead to the failure of the component therefore, in many circumstances such kind of very localized corrosion is more detrimental than uniform corrosion.

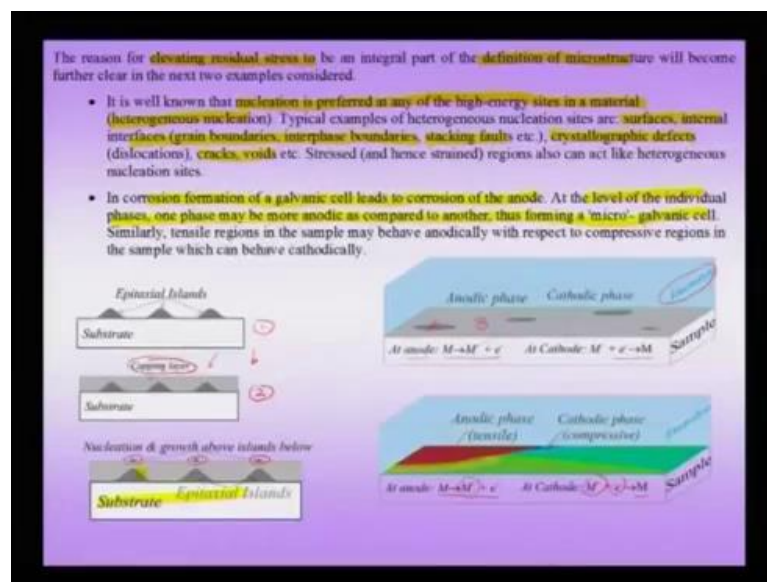
This can actually be planned for and which you can plan a replacement or what is called preventive maintenance replacement, but can we have electro chemical corrosion occurring because of residual stresses, the answer is yes. In this case, what happens is that regions which are tensile in the material and the diagram, you can see below here there is a region.

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For instance, we considered the precipitate here and we saw there are regions which are tensile as compared to the remaining region which could be compressive, so for instance this is now my compressive region and this is my tensile region.

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So, suppose I take as a schematic example, a region which is tensile here and a region which is compressive here, now you can actually form an electro chemical or galvanic cell between the tensile and compressive region. The tensile region may play the role of an anode here wherein the metal gives rise to an impulse and an electron that means it

dissolves into the electrolyte and at the cathode the positive ion combines with the electron. In other words, now I can have a selective dissolution of the anodic tensile region this is occurring of course, at the scale of length scale of micro structure.

I am showing here, but this is occurring not because of course, it could be because of the as we saw the precipitate in the previous case, but it could also occur purely because of the residual stresses present in the material. We have just taken forsake certain distribution of residual stresses here, but they could be associated with for instance thermal stability. It could be across an interphase or could be even be a dislocation etcetera there, but important point is that now selective dissolution of the anode or the anodic region, which is tensile in nature.

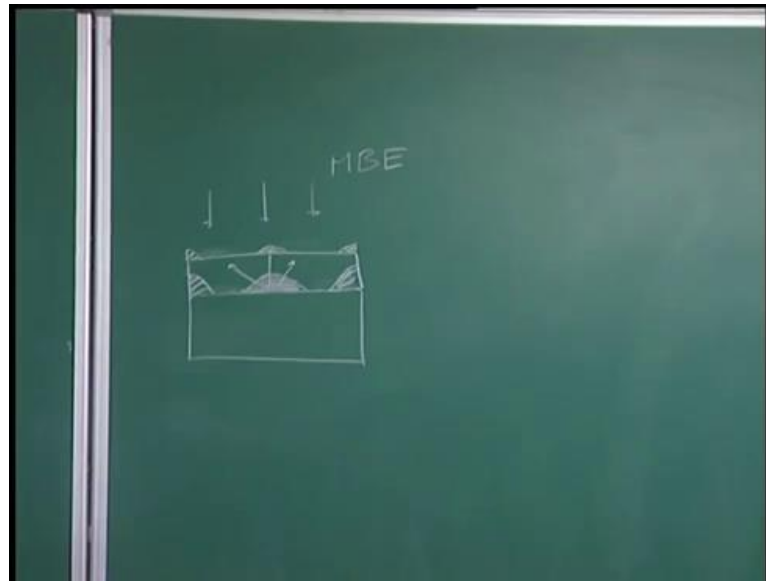
It is not a different phase or not a different electrode as one normally conceives in a macroscopic galvanic scenario. Therefore, clearly from these two examples, which we have considered, now one being the case of hetero epitaxial growth of islands.

More importantly, hetero epitaxial growth of buried islands or the second layer or the case of galvanic corrosion that residual stresses can play a very important role. They need to be actually elevated to the level of phases and their distribution or of the level of defects and their distribution. Therefore, a comprehensive definition of micro structure should involve phases defects and residual stresses and of course,, their distributions and these two examples are very important in exemplifying this important aspect, so Anil has a question.

Student: In the figure, they say not able to grow then what happened.

Good question here, so in the case we have just talked about we talked about epitaxial islands, but it is not necessary that islands be epitaxial. For instance, I could take a glass substrate and I could grow a crystalline layer on it, which could be for instance a gold layer on top of it. This gold layer of course, may form some kind of an what you may call a spherical body or a body which is in a certain way, you know deposited on this, there is clearly an absence of any epitaxial stresses.

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There is the whole origin of stresses in this case is the need for the system is to match atoms or match atomic planes from the epitaxial lower layer with the substrate. Why does the system want to do this, the system wants to do this because if there is an epitaxial interphase or a coherent interphase, the interphasial energy cost for the system is small. In the nucleation stage, we know from the nucleation theory that interphasial energy is playing a very important role in nucleation stage. Therefore, when the precipitate is small it is nucleating on the surface it wants to choose that configuration which can minimize the interphasial energy.

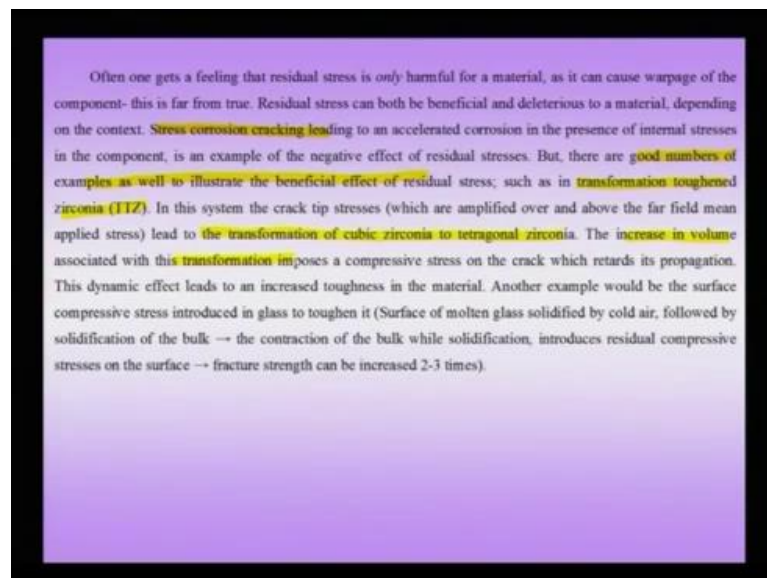
What is the cost it pays the system pays, a cost in terms of the coherency stresses, so there is a volumetric term which is the coherency stresses which is the additive term in other words it is opposing the nucleation here. There is an interphasial energy term which is low and therefore, it prefers to have a coherent interphase and later on when the system evolves with time, you will notice that what might happen is that there may be interphasial misfit dislocations. This form, which is actually partially relieve my coherency stresses or the epitaxial stresses and at even larger growth.

You may notice that this interphase may actually become completely incoherent because of presence of many dislocations here. So, in case where epitaxial system that means in glass substrate, then this kind of preferential alignment is not expected unless of course, again if there is some epitaxial ability in the over layer or the film. There is some origin

of the residual stresses which can give rise to this alignment because this kind of an alignment can be preferred larger the anisotropy of the system.

In other words, the over layers which are grown on it, so let us talk about another important point with respect to residual stresses in a material and this comes from often from the engineer application of the materials that we think. We see that whenever there are residual stresses there is a warping of a component and often there is a feeling that this is this implies that residual stresses are harmful for the material. We also see from processes like short preening that actually that there can beneficial effects for residual stresses. So, we should understand that residual stresses can be beneficial or detrimental depending on the situation, so we will take an example of both.

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So, let us talk about you know when we are talking about stress corrosion cracking which is a accelerated form of corrosion in the presence of internal stresses in the material or the component. In this case, clearly the residual stresses are bad, so because that is what is giving rise to this what you might call stress corrosion cracking. In the absence of stresses, this would be normal corrosion and that would be much which will have a much lower impact or the negative impact on the material.

So, clearly there are reasons to believe that residual stresses can be bad like in the case of stress corrosion capping or warp age of a component etcetera. There are a good number of examples which we can talk about in what you call to illustrate the beneficial effect of

residual stresses. We can consider a couple of them in the current slide when we are talking about the transformation toughened zirconium. So, what happens in this case that the crack tip stresses and we saw that crack tip stresses themselves of course, crack tips have no stresses.

They tend to amplify far field stresses, so we have some sort of a co opted crack tips into definition of residual stresses. So, the crack tip stresses which amplify the stresses and this high stress value leads to the transformation of cubic zirconium to tetragonal zirconium.

In transformation, toughened zirconium, the zirconium is present in the meta stable form and because of the presence of these stresses the zirconium transforms from the cubic form to the tetragonal form and of an also to the monoclinic form. This leads to an increase in volume associated with the phase transformation, this increased volume imposes a compressive stress on the crack tip.

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So, cracks can only open in the presence of tensile stresses, so I am applying a mean field far field stress and at the head of the crack tip material like this. For instance, this is my material I see that the crack tip can actually amplify my stresses, I am now assuming a sharp crack. Now, if the region at the head of the crack tip is actually transforming say suppose this region because of this high presence of high stress actually transforms, so this is my region of transformation.

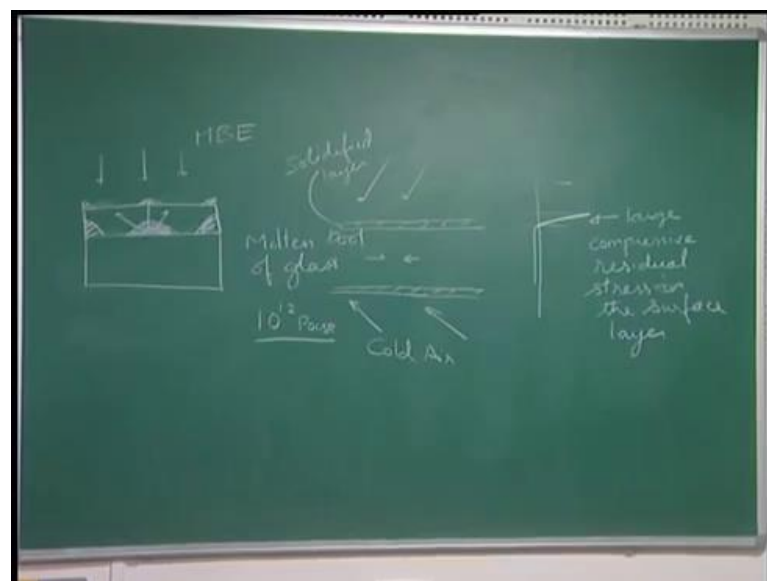


So, this volume is larger because the presence of this tetragonal zirconium and monoclinic zirconium and this imposes a kind of compressive or closing stress on the crack tip. This is what I observe is that this material actually gets toughened because if there were no transformation happening and zirconium being a brittle material, this little ceramic the crack would propagate and lead to failure of the material, but because of this phase transformation.

You can see that this is actually giving rise to toughening of the material, so this is some kind of a secondary effect coming from crack tip stresses, but the direct effect is the next one which we are going to consider which is example of toughened glass. So, we know that glass is an amorphous material and here we are talking about silicate glass a normal glass which we have in window panes etcetera. Now, because being a very brittle material in spite of its beautiful transparency etcetera, it has certain limited applications.

Now, we also know that there are glasses like many in many cases which are actually impact resistant, we can talk about bullet proof glass etcetera. So, what is the origin of the toughness of these forms of glasses and we will consider one way of actually toughening glasses, which is putting residual stresses into this glass, so what happens is that let me consider a molten pool of glass here.

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So, I can allow this molten pool of glass to solidify normally, but instead of doing that, I blow cold air from the outside and because of this thin crust of glass first solidifies. After

this of course, I stop blowing and allow my entire glass volume to form a solid state of course, we should note that glass being amorphous; it does not display a strong melting point. Nevertheless, its viscosity increases to a certain level about may be about 10 power 12 Poise, where it can be considered as a solid.

Now, what happens when the remaining of the pool of the glass tries to solidify, it will contract because the liquid, the solid has a lower volume as compared to the liquid. Therefore, when it is trying to contract this glass as it is solidifying the outer regions or the upper outer crust is actually already solidified. Therefore, what will happen, it will try to pull in this region and thus impose compressive residual stresses on the surface. So, if I look at the residual stress plot, I would notice that close to the surface, you will actually have large compressive stresses, this is a negative side compressive side and of course, there will be small.

So, if I plot my residual stresses, we have a large compressive residual stress on the surface which is which will stop actually crack propagation from the surfaces. We are talking about the glasses, we should note that the on the surface twice as deleterious as crack on the interior and whenever of course, there are bodies tried to be deformed and bending the maximum stresses occur on the surface. Therefore, from two points of view from the fact in certain kinds of loading the certain stresses occur on the surface number two.

From the point of view that the cracks on the surface are actually twice as deleterious as cracks on the interior. So, suppose I had cracks of length  $a$  on the surface, this would be as bad as a crack of length  $2a$  on the interior. Therefore, I need to keep my surface as compressively loaded as possible and that is what is being done by this process of selectively cooling this and solidifying the surface first. Then, cooling the remaining of the interior to give rise to a glass, so the composition has not been changed in this case the phase distribution has not been changed in this case what has we played around with is the presence of residual stresses.

As we know, residual stresses are always 0, overall in the body given the formula for the integral  $\sigma dv$  equal to 0. Therefore, the outer is in compressive stresses, there is a small residual internal tensile stress in the middle of the body. This large compressive residual stresses actually give rise to the large strengthening or toughening of the glass

which can be three to four times of can be even higher in many cases. Therefore, now playing or engineering my residual stresses I am able to obtain large toughness in the glass.

Therefore, it is very clear that residual stresses are very important and they are to be considered as a part of the functional definition of micro structure. As we will see, as we go into the study of nano materials, we will note that each one of these components of the definition of micro structure. For instance, the phases or the defects distribution defect structure and the residual stress all could get into the nanoscale, and therefore in nonmaterial, all of them would have an equally important or if not a more important role to play in determining the properties. Of course, the nanostructure or the nano material and also the entire component on the whole is made of such a structure or a material.

Now, we could of course talk about a certain length scale, we could talk about properties at that length scale, but more important is that how we go from one length scale to other length scale to even larger length scale. Finally, how each length scale talks to each other giving rise to what you might call what we might usually observe as macroscopic property. In other words, it is not enough that I reside inside a certain length scale and worry about the properties at that length scale, I need to see how this length scale talk to next higher length scale.

How that length scale talks to next higher length scale and how are properties varying across these length scales, finally giving rise to what we might think as a property or the performance of an entire component. So, just to take up a couple of examples that how we can talk about what we are traversing across length scales.

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**1 Change of properties across length scales: polycrystalline copper (CCP structure)**

Atomic level ( $\text{\AA}$ )  $\rightarrow$  Unit Cell level (few  $\text{\AA}$ -nm)  $\rightarrow$  Grain level (nm- $\mu\text{m}$ )  $\rightarrow$  Material level (cm)

At the atomic level there is order only in the average sense (at  $T > 0\text{K}$ ) as the atoms are constantly vibrating about the mean lattice position. Hence, in a strict sense the perfect order is missing (a). The unit cell level is the level where the atomic arrangement becomes evident (crystal structure develops) and concepts like Burgers vector emerge. b. It is at this level that averaging with respect to probabilistic occupation of lattice positions in disordered alloys is made (say  $\text{Ni}_{50}\text{Al}_{50}$  alloy is defined by a 50-50 probability of Ni or Al occupying a lattice position). At the grain level (c, which is a single crystal), there is nearly perfect order (as the scale of atomic vibrations are too small compared to grain scale), except for the presence of defects like vacancies, dislocations etc. At this scale the material is also anisotropic (e.g. with respect to the elastic stiffness, which is represented by three independent numbers:  $E_{11}$ ,  $E_{33}$  &  $E_{44}$ ). It is to be noted that the Cu crystal may be isotropic with respect to other properties. At the material level (d), assuming that the grains are randomly oriented, there is an averaging of the elastic moduli and the material becomes isotropic. At this scale, the crystalline order which was developed at the grain level (c) is destroyed at the grain boundaries and there is no long range order across the sample. When the material is rolled or extruded, it will develop a *texture* (preferred directional properties), which arises due to partial reorientation of the grains. That is, we have recovered some of the inherent anisotropy at the grain scale. As we can see, concepts often get 'inverted' as we go from one length scale to another.

(a) Thermal vibration (b) Unit cell of Cu (c) Grain Structure (d) Sample of Cu

We may take two simple common examples and we will talk about different interesting aspects which come about when you do traversing of length scale. So, we talk about polycrystalline copper as our first example.

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**2 Change of properties across length scales: Fe sample which has not been magnetized**

Atomic level ( $\text{\AA}$ )  $\rightarrow$  Domain level (few  $\mu\text{m}$ )  $\rightarrow$  Material level (cm)

Consider a magnetic material (E.g. Fe, Ni) below the Curie temperature (but  $T > 0\text{K}$ ), where it is ferromagnetic in nature. In this condition the atomic magnetic moments try to align, but thermal effects lead to partial disordering. This takes place within regions in the sample called domains which are typically of micrometer size. The configuration of the domains is in such manner so as to reduce the magnetostatic energy. This arrangement of domains, wherein they are not preferentially aligned, leads to *no* net magnetization of the sample. Hence the story as we traverse length scales is:

Atomic magnetic moments ( $m_{\text{atomic}}$ )  
 $\rightarrow$  Less magnetization in a domain than the number of atomic moments (domain) (say if  $n$  atoms are there, then the net magnetic moment within a domain  $\neq n \times m_{\text{atomic}}$ , turns out to be less than  $n \times m_{\text{atomic}}$ )  
 $\rightarrow$  No net magnetization at the sample level.

Electron Spin, Nuclear Spin, Closure Domains

As a second example, we will take up the example of what you call an iron sample which has been magnetized.

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**Change of properties across length scales: polycrystalline copper (CCP structure)**

Atomic level ( $\text{\AA}$ )  $\rightarrow$  Unit Cell level (few  $\text{\AA}$ )  $\rightarrow$  Grain level (nm- $\mu$ m)  $\rightarrow$  Material level (cm)

At the atomic level there is order only in the average sense (at  $T > 0\text{K}$ ) as the atoms are constantly vibrating about the mean lattice position. Hence, in a strict sense the perfect order is missing (a). The unit cell level is the level where the atomic arrangement becomes evident (crystal structure develops) and concepts like Burgers vector emerge. It is at this level that averaging with respect to probabilistic occupation of lattice positions in disordered alloys is made (say  $\text{Ni}_{50}\text{Al}_{50}$  alloy is defined by a 50-50 probability of Ni or Al occupying a lattice position). At the grain level (c, which is a single crystal), there is nearly perfect order (as the scale of atomic vibrations are too small compared to grain scale), except for the presence of defects like vacancies, dislocations etc. At this scale the material is also anisotropic (e.g. with respect to the elastic stiffness, which is represented by three independent numbers:  $E_{11}$ ,  $E_{22}$ , &  $E_{33}$ ). It is to be noted that the Cu crystal may be isotropic with respect to other properties. At the material level (d), assuming that the grains are randomly oriented, there is an averaging of the elastic moduli and the material becomes isotropic. At this scale, the crystalline order which was developed at the grain level (c) is destroyed at the grain boundaries and there is no long range order across the sample. When the material is rolled or extruded, it will develop a texture (preferred directional properties), which arises due to partial reorientation of the grains. That is, we have recovered some of the inherent anisotropy at the grain scale. As we can see, concepts often get "inverted" as we go from one length scale to another.



(a) Thermal vibration (b) Unit cell of Cu (c) Grain Structure (d) Sample of Cu

So, we will take up two examples to understand how we are going to traverse across length scales. So, what is the first length scale I need to talk about in the case of a copper polycrystal the first length scale is of course, an atomic length scale. If you note that in an atomic length scale at any finite temperatures atoms are actually vibrating they are not stationary. That means there is a lot of disorder at the fundamental atomic length scale, therefore at the atomic level, there is order only in the average sense at any temperature. Typically, we are always talking about temperatures which are greater than 0 Kelvin, atoms are constantly vibrating about their mean lattice position.

Of course, we have not yet come to the lattice scale they are vibrating about some kind of a mean position hence in a stricter sense perfect order is missing. At the next length scale, which is length scale of what you might call the unit cell length scale level, the atomic arrangement becomes evident that is the crystalline. The crystal structure develops, so here I divide the problem into the four length scales the atomic level, which is at the angstrom length scales the unit cell level. If I am talking about simple crystal structures like copper, I have to talk only about a few angstroms, but there could be other crystal structures, which have a much larger lattice parameter.

They could be in the nanometer length scale, but typically it is between about a few angstroms to a few nanometers typically though we could always talk about crystals which have much larger lattice parameter. The next level which I am talking about which

I will talk about is the level of the grain or the level of the single crystalline part of a poly crystalline specimen. Typically, this is of the order of microns, of course special, we can see that these grains size can actually be reduced to nanometer, which is one of the things we will consider during this course. How can I produce nano crystalline copper and what are the importance or advantages of producing the nano crystalline materials or what you may call nano structured materials.

When we jump up from the micron scale to the scale of the material or the scale of the entire component, now we are talking about centimeters or even larger. Then, we shall see how the properties have come a few orders of magnitude in terms of dimensions of angstroms to a centimeter which is about  $10^7$  magnitude and how I can actually see various kinds of phenomenon flipping from. I am talking about order how order flips from highly disordered state or a average order sense to certain other kinds of configurations.

So, at the unit cell level, where atomic arrangement becomes evident to us that means we are actually seeing a crystal structure developed concepts like buggers vectors emerge. So, when I am talking about buggers vector, I am actually using my unit cell as the fundamental definition and it is a buggers vector is nothing but a fundamental lattice translation vector or the shortest lattice translation vector for a perfect dislocation. It is at this level I actually do an averaging with respect to say probabilistic occupation. For instance suppose I am talking about a disordered alloy, which is say for instance nickel 50, aluminum 50 alloy and that means that there are 50 percent of atoms in the system which are of the type of nickel and 50 percent of the type of aluminum.

Of course, for the simplicity, let me talk about the kind of the structure wherein the body, there is a body centre and there is a phase centre and we are not limiting ourselves to the example of poly crystalline copper. We are actually digressing a little bit to an alternate example just to tell us that where does the averaging with respect to composition takes place it takes place in at this level of the unit cell. So, in an Ni Al system with a 50, 50 possibility, if I take a single unit cell, I may notice that it may not contain 50 percent atoms of nickel or aluminum.

When I take a few unit cells or larger cluster of unit cells and average out there, then I would notice that 50 percent of the atoms are nickel and 50 percent of the atoms are

aluminum. If it is a disordered alloy, then I would notice that there is no preferential occupation of either aluminum or nickel in any of the sites. So, it could be randomly be positioned anywhere, so first I have the level of the atomic scale, wherein there is no perfect order, it is actually the atoms are vibrating about their mean position, when I go to the next length scale with a length scale of the unit cell.

I am talking about a pure material, I would notice that there is some sort of a crystalline order. That means the atoms are positioned close to their lattice position, in other words I could not worry about the small oscillations, which are present at the atomic length scales. If I am talking about a few unit cells, then I may see that these oscillations are much smaller than what you might call a few lattice parameters length scale. Also, I noticed that at this length scale there could be a disorder of different type, the disorder with respect to for instance with respect to a probabilistic occupation of atoms or the compositional averaging which I need to do.

The next level which is not just the length scale of a few unit cell, but the length scale of a single grain and you can see this micro structure here, which is shown for a pure copper. There are many grains and within these grains, you may observe interesting features like these are twin boundaries here and I could restrict myself to this bright region which is a single grain of copper. Now, this single grain of copper of course, is random with respect to its neighboring grains and there is no preferential alignment. I hope with respect to the neighboring grains at this grain level, which is a single crystalline part of this poly crystalline material.

There is nearly perfect order because the scale of atomic vibration is too small as compared to the scale of the entire grain. The scale of entire grain could be in the order of for instance order of a 100 microns, while atomic vibrations are less than the lattice parameter. Therefore, at this length scale I can think of this system as nearly perfect with respect to my thermal vibration or the disordering, which is coming from thermal vibration. However, at this length scale I may observe that there are crystallographic defects like there could be dislocations, there could be vacancies, there could micro cracks etcetera, there could be even stacking faults or as in the case we have seen here a twin boundary.

Therefore, even though with respect to thermal vibration, there is some sort of an order at this length scale, but there is no perfect order even at this length scale from the perspective of defects like crystallographic defects in the material. At this scale, the material is also anisotropic, for instance it could be anisotropic with respect to elastic stiffness and if I were to talk about the elastic properties of this body, actually I will have to feed in three independent elastic module. They typically called  $e_{11}$ ,  $e_{12}$  and  $e_{44}$ , if I want to capture the elastic behavior of the material at this length scale.

Therefore, the material is anisotropic that means there is a direction dependent to its properties at the length scale of a grain. However, the same single crystal could also be isotropic with respect to some other properties. For example, with respect to its optical properties, but definitely it is anisotropic with respect to its elastic properties. Next length scale which we need to talk about is the length scale of the entire sample for instance here we have seen a photo of a copper sample at this length scale. You will notice that the material is actually isotropic and the reason it is isotropic is that because the grains which are anisotropic are now randomly oriented in space.

Therefore, when I am averaging over all these random orientations and we see that there is no preferred orientation to this elastic moduli and I can describe such a material in terms of just two moduli that is  $e$  and  $\nu$ . Of course, I can use  $G$  and  $\nu$  or one of the other two moduli, therefore I just need two elastic moduli to describe the entire polycrystalline copper specimen. So, there is no preferred orientation at this length scale, however if I roll or extrude this material, I would notice that I can develop a texture in this material. In other words, it could actually develop a preferred orientation now and which occurs due to the partial reorientation of the grains.

Of course, the kind of the texture which develops depends on the deformation of the process which is employed, but now in the presence of texture the material again develops another kind anisotropy. In other words, we have retrieved some of the anisotropy which was present at the grain level, so it is one of the anisotropy which was present at the grain level. So, it is one of the crystallographic anisotropy, which was present which was actually we have retrieved. Therefore, we can see that concepts often get inverted as we go from one length scale to another and how properties change actually go from one length scale to the other.



Therefore, it is very important that I understand my material behavior material properties and material structure by traversing across entire length scales to summarize this slide we have just now seen. We will notice that when we are at the length scale of the individual atom, there is lot of disorder, there is thermal vibration at the length scale of a few unit cells. We see that crystalline order has developed and when I am talking about say few tens of unit cells the atomic vibration is much smaller as compared to that length scale. Definitely at the grain length scale, I see that the things are completely ordered with respect to this thermal vibration.

However, at the thermal length scale, we will notice there are other defects which come into play which are crystallographic defects like dislocations voids etcetera or even stacking faults or twin photo micro graph shows here. Therefore, again I am dealing with certain other kind of disorder at this length scale. Now, if I am talking about the single unit cell level, I would notice that my system has to be described by anisotropic properties which are what is seen at the single grain level. When I go to an entire component level, I see that now my material behaves isotropically unless of course, it has some kind of a texture.

So, a material typically with random oriented grains is completely isotropic and at this length scale, I do have to worry about some amount about the crystallographic defects present in the material. More importantly, also I have to worry about the defects which arise between the grains. For instance, now I need to worry about the grain boundary in the material, so as I am going to different length scales, I worry about different issues which finally, affect my properties. Now, for instance the grain boundaries would imply that if I am talking about a single crystal and presence of dislocations, the material would be weak to a certain extent.

Suppose, the dislocations want to move grain boundaries would typically provide an impediment to the motion of dislocations and therefore, we have hardening which we often refer to hall patch kind of an hardening at the entire material level. Now, I have only to describe the whole properties for instance the elastic property, which is two independent moduli unlike the case of the grain, wherein I have to use or a single crystal where I have to use three independent moduli.

So, it is clear that when I want to understand any property, I want to understand structure, I want to therefore deduce the performance of a component, which is made from a material like which is shown here. I have to traverse across length scales, it is not essential that it is not enough that I worry about only one length scale. Of course, I have to talk about various proings which gives me the effect of those proings and the proings parameters on the evolution of the material at these various length scales.

For instance, suppose I am doing a heat treatment there are certain temperatures for the material, if I am talking about for instance solid solution of nickel and aluminum it could be disordered, but certain other temperature it could get ordered. Typically, it would get ordered lower temperatures and this ordering can actually alter my crystal structure and if it alters my crystal structure, it is going to alter my properties my concepts like buggers vectors. It is also going to alter my entire set of properties which arise in length scales above the crystalline level.

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2 Change of properties across length scales: **Fe sample which has not been magnetized**

Atomic level (Å) → Domain level (~few μm) → Material level (cm)

Consider a magnetic material (E.g. Fe, Ni) below the Curie temperature (but  $T > 0K$ ), where it is ferromagnetic in nature. In this condition the atomic magnetic moments try to align, but thermal effects lead to partial disordering. This takes place within regions in the sample called domains which are typically of micrometer size. The configuration of the domains is in such manner so as to reduce the magnetostatic energy. This arrangement of domains, wherein they are not preferentially aligned, leads to *no* net magnetization of the sample. Hence the story as we traverse length scales is:

Atomic magnetic moments ( $m_{atomic}$ )

- Less magnetization in a domain than the number of atomic moments (domain) (say if  $n$  atoms are there, then the net magnetic moment within a domain  $\neq n \times m_{atomic}$ , turns out to be less than  $n \times m_{atomic}$ )
- No net magnetization at the sample level.

Now, we are in a position to actually proceed to the second example, which the example of an iron sample which has been magnetized.