

Advanced Materials and Processes
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Lecture – 12
Bulk Metallic Glass, Glassy and Amorphous Materials (Contd.)

Welcome to NPTEL myself Dr. Jayanta Das from Department of Metallurgical and Materials Engineering, IIT Kharagpur. I will be teaching you Advanced Materials and Process. In last couple of classes we have discussed about the glass forming ability based on two different aspects; one is the topology of the short range order which was discussed by several scientist and the other one was empirical relations.

The empirical relations has been developed based on some of the characteristic temperature like glass transition temperature, crystallization temperature, liquidus temperature and so on. And those equations we have seen are very much valid in order to represent the glass forming ability.

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The slide is titled "Glass forming ability (GFA): Physical Properties" in red and blue text. A red arrow points to the title. Below the title, a white box with a blue border contains a list of physical properties, each preceded by a red checkmark:

- ✓ - Minimum Molar Volume
- ✓ - Viscosity and Specific Volume
- Other Parameters
 - ✓ - Enthalpy of Vacancy formation
 - ✓ - Local Ordering
 - ✓ - Fragility Parameter

The slide also features a small video inset of the lecturer in the bottom right corner. At the bottom, there are logos for IIT Kharagpur and NPTEL Online Certification Courses, along with the text "Bulk Metallic Glasses by C Inoue, CRC Press, 2011".

And today we will continue that discussion and we would like to see that how different physical properties are correlated with the glass forming ability of the alloy. So, along this line we find out these physical properties like the minimum molar volume and the viscosity of the liquid is also important in order to explain a good or bad glass forming ability of a designed alloy.

And there are also some other parameters people or scientists have correlated. Those are enthalpy of vacancy formation. Let us say the local ordering means we are talking about not long range order the ordering inside short range order; short range order means basically less than 2 nm in size and also some other parameter like fragility parameter.

And today we will discuss what are they? What is the fragility? What we really mean by the viscosity of a under cooled liquid and so on. So, in case of a minimum molar volume there are some equation that came out and it says that the misfit energy is important.

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Glass forming ability (GFA): Minimum Molar Volume

The misfit energy in a liquid and the atomic volumes and isothermal compressibility of the constituent elements are important:

The critical composition in a binary alloy system at which the decrease in mean atomic volume is the highest (at which it is also expected to have the highest viscosity)

$$X_B = \frac{1}{1 + \sqrt{\frac{V_A \chi_A}{V_B \chi_B}}}$$

X_B represents the critical solute concentration
 V_A and V_B are the atomic volumes of elements A (solvent) and B (solute), respectively
 χ_A and χ_B are the isothermal compressibilities of pure A and pure B, respectively, in their liquid states

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So, in a liquid the misfit energy and the atomic volume of the isothermal compressibility of this constituent element has somehow very much important. What does it mean that a critical composition in case of a binary alloy if you think about a binary metallic glass, there the decrease in the atomic volume is the highest at which it is also expected to have the highest viscosity.

So, you can see that these relations are very much similar and correlated to the one which we had discussed in the last class with the atomic mismatch. So, if we think about X_B which is the concentration of the solute concentration of B element, and let us say V_A and V_B are the atomic volume then we can represent that X_B which is the minimum or the critical solute concentration requires in order to form a glass. So, χ_A and χ_B they are basically isothermal compressibility.

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Glass forming ability (GFA): Viscosity

The viscosity of the best glass-forming alloy was an order of magnitude higher compared to the worst glass-former at their respective liquidus temperatures.

The best glass-forming alloy showed the smallest volume change upon crystallization and the worst glass-former showed the largest change.

The viscosity and correspondingly small free volume in the liquid at the melting temperature and contribute significantly to improve the GFA of alloys.

VFT Equation (Vogel-Fulcher-Tammann)

$$\eta(T) = \eta_0 \exp\left(\frac{D^* T_0}{T - T_0}\right)$$

T_0 is referred to as the VFT temperature
 D^* is called the "fragility parameter"
 η_0 is the high temperature limit of viscosity

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Now, there are also another one very major aspect that the physical properties that one should think about, that is basically the viscosity. Because we understood that the viscosity of a liquid continuously decreases and when it reaches to 10^{12} Pa/s or greater than 10^{12} - 10^{13} Pa/s and then it basically give you the glass ok, so it is basically the solid viscosity.

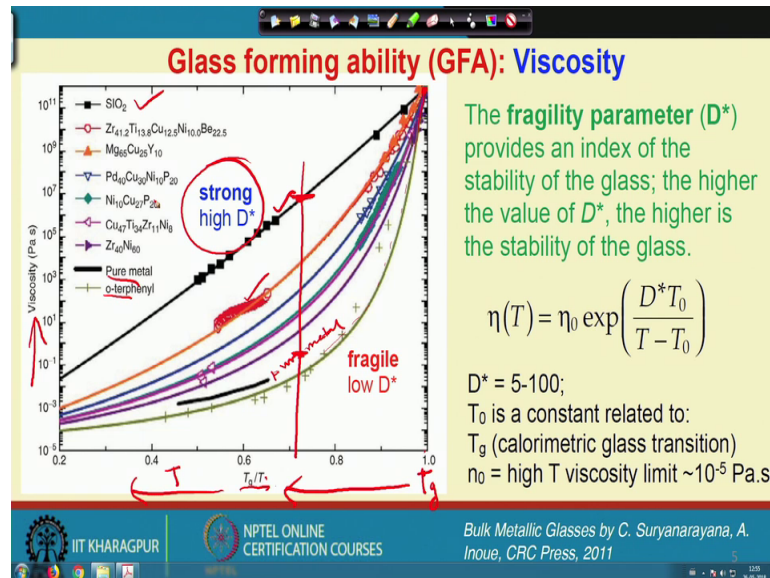
So, a best glass forming alloy will always show a higher liquid viscosity almost an order of magnitude higher than the worst glass former. And also the best glass forming alloy show the smallest volume change upon crystallization ok. So, the worst glass former always show a larger volume change.

So, if you see the plot of specific volume versus temperature we will understand that what is the meaning of the excess volume; ok. So, the viscosity and correspondingly small free volume in the liquid are at the melting temperature contribute significantly to improve the glass forming ability. However, these understanding require some mathematical expression.

And the Vogel-Fulcher-Tammann they basically described these like Arrhenius type of equation where the viscosity at a given temperature is basically the high temperature limit of the viscosity and D^* here is referred to a fragility parameter and T_0 is referred to a VFT temperature.

So, these two appear to be constant for a given alloy ok. So, it says that that any given temperature the higher the viscosity of an alloy will always show you a higher glass forming ability. And to understand this what we need to do we need to look at what are the different viscosity values of a good glass former and a worst glass former and we need to understand that.

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Please have a look at the left hand side figure here it is a normalized temperature, it says that if we increase the temperature from a glass transition temperature this is basically T_g and we are going towards the melting temperatures. So, T is increasing here ok, so T is increasing here. So, here is the T_g when T is the T_g then it has a value of 1.

So, here is basically the glass transition has happened and above glass transition we are measuring the viscosity of the super cooled liquid ok. And then we are plotting here the viscosity versus the normalized temperature. And this is let us say the viscosity change with temperature of some polymer of some pure metal, this is the pure metal which is a worst glass former ok.

And now this one is Vitreloy which is one of the best glass former in metallic system. And this square bubbles are the silicon dioxide which is one of the best glass possible in oxide system ok. So, you see that the best glass former always show the best glass former always show a viscosity level which is much higher at any given normalized temperature. So, the viscosity is very high here and the viscosity of the pure metal is low

here, or the viscosity change viscosity decreases very abruptly in case of a worst glass forming alloys or a or material ok.

So, we can categorize this side of the plot as a strong glass former which will depict a higher value of D and here are the fragile, or worst glass former. So, here this fragile terminology does not mean it is a mechanical fragility this is a kinetic fragility we are talking about. So, here this fragility parameter D^* provides an index of the stability of the glass.

So, here one thing is very much interesting you see that we are measuring the viscosity of the super cool liquid, and we are describing the stability of the glass which is which really happen below the glass transition temperature. We transform from the super cool liquid to a glass. So, these two are very much interlined and the D^* usually has a value of 5 to 100.

So, let us say higher than value the higher or stronger the glass and T_0 is a constant, which is related to a calorimetric glass transition. So, T_g is basically the calorimetric glass transition, and definitely the it has 0 is the high temperature viscosity which has somewhat value of 10^{-5} pas.s.

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Glass forming ability (GFA): Other parameters

Enthalpy of Vacancy formation

- measurement can be done using electrical resistivity
- GFA increase - ΔH_v , ΔH_{mix} , ΔS_{config} decreases

Local Ordering

- some metallic glasses show strong tendency of "chemical short range order" SRO
- Icosahedral SRO
- $Cu_{70}Hf_{30}$

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So, the viscosity appears to be very much important, the viscosity of the under cooling liquid is very much important to illustrate the glass forming ability. Now, there are

several other parameter has been has been discussed and told to linked with the glass forming ability like the enthalpy of a vacancy formation.

What it said that the measurement of vacancy formation in a glass can be done using simple electrical resistivity measurement ok. And the glass forming ability will increase in a case when the enthalpy of vacancy formation, the enthalpy of mixing, and the configuration the entropy will decreases ok.

So, definitely the stability of the glass will increase and also the glass forming ability will also increase. What I want to mean that if we introduce a defect ok. So, the vacancy is a defect and then definitely we will get a different configuration ok. And then by using that configuration we can decrease this value.

Now, in some cases it has observed that the local ordering in the short range order is also linked with the better glass forming ability. In some cases it has been observed that he chemical short range order is also important. What I want to mean that I have two different type of atoms which is making that short range order. And so whether a preferred a chemistry is required in that short range order to give the stability.

Now, let us say a particular type of short range order in terms of structure icosahedral structure of the short range order which has been observed. Let us say in case of a binary copper hafnium glass is also gives us a better stability, and better glass forming ability. So, these are some of the other parameter which appears to be important in some of the glass forming alloy system.

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Glass forming ability (GFA): Other parameters
Fragility Parameter

Prof. Angell classified liquids as “strong” or “fragile”

- Good glass-forming alloys are strong liquids with a high viscosity, while the fragile liquids are only marginal glass formers.
- GFA can be a complex function of fragility, T_{rg} and T_x. $T_{rg} = \frac{T_g}{T_l}$
- The fragility provides a measure of the sensitivity of the structure of a liquid to temperature changes.

Fragility index

$$m = \frac{d[\ln \tau(T)]}{d(T_g/T)} \Big|_{T_g}$$
 $\tau(T)$ is the temperature-dependent relaxation time
 T_g is the glass transition temperature

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Also, let us say Professor Angell who basically mostly done researches on this viscosity and a strong and fragile liquid. And he classified this idea of strong and fragile glass forming liquid, and he classified these two different type of liquid, in one case which has a strong glass former means higher glass forming ability and poor glass forming ability.

So, the strong liquid with a very high viscosity and the fragile liquids are very marginal glass former that we have already talked. And so we can think about the glass forming ability of a complex function of the fragility and T_{rg} that is the reduced glass transition temperature, and the T_x because we have seen that $T_{rg} = \frac{T_g}{T_l}$.

So, T_x, T_{rg} these are all these empirical relation came out and we now introduce another term that is the fragility based on the viscosity. So, the fragility means that a strong, or fragile glass former and how easy glass formation could be provided. So, here the fragility provides a measure of the sensitivity of the structure, or the short range order, or the cluster we are talking about.

So, the physical significance of fragility gives you the sensitivity of the structure of a liquid with the temperature changes ok. So, if the structure changes very easily or kinetically it is more favor then it is a very poor glass forming alloy ok. Because in the earlier plot we have seen that same situation once again I would like to show you the earlier plot.

So, here also this is the same that a poor glass forming alloy has a very fast change in the viscosity means the structure of that short range order changes very fast ok.

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Glass forming ability (GFA): Other parameters
Fragility Parameter

Prof. Angell classified liquids as “strong” or “fragile”

- Good glass-forming alloys are **strong liquids** with a **high viscosity**, while the **fragile liquids** are only marginal glass formers.
- GFA can be a complex function of **fragility, T_g and T_x** .
- The fragility provides a measure of the **sensitivity of the structure of a liquid** to temperature changes,

Fragility index $m = \frac{d[\ln \tau(T)]}{d(T_g/T)} \Big|_{T_g}$ $\tau(T)$ is the temperature-dependent relaxation time
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In the glass forming where this is a more stable glass. Now, in this case, Prof Angell introduces a Fragility index which is very similar like D star ok. So, in this particular case he told that the change of the temperature dependent relaxation time which can be measured directly using differential scanning calorimeter. And the glass transition temperature with change in the temperature could be classified and can be measure of a fragility index.

And from there, we can directly say whether the glass is a better glass forming alloy or a in or a poor glass forming alloy like the fragility parameter, or the fragility index we have discussed earlier.

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Glass forming ability (GFA): Other parameters

Mechanical alloying (MA)

A large number of crystal defects and lattice strain are introduced upon heavy deformation experienced by the powder particles during MA, .

These defects raise the free energy of the crystalline phase to a level above that of the hypothetical amorphous phase.

Consequently, the crystalline lattice becomes destabilised and the amorphous phase becomes stabilized.

$$\Delta G_c + \Delta G_d > \Delta G_a$$

- ✓ ΔG_c is the free energy of the crystalline lattice
- ✓ ΔG_d is the increase in free energy due to incorporation of crystal defects
- ✓ ΔG_a is the free energy of the amorphous phase

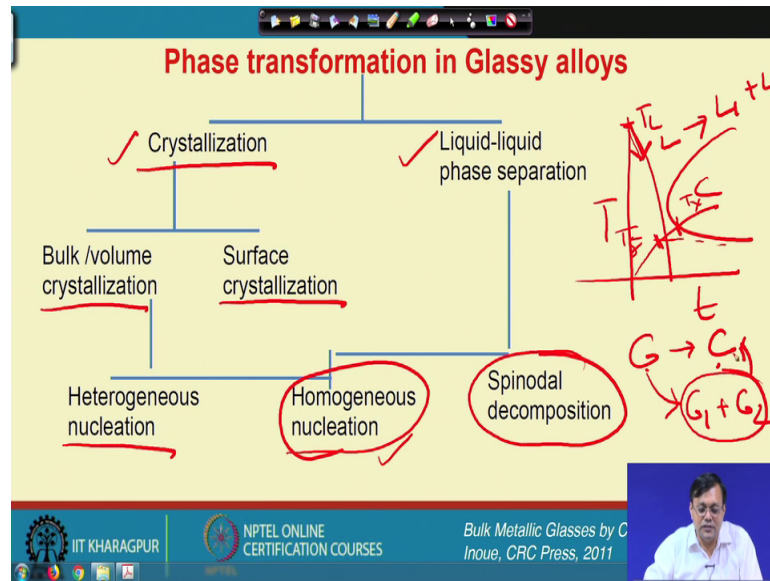
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Now, there are definitely some other parameter including the mechanical alloying, so many of the scientists they have prepared glassy alloys by mechanical alloying or let us say ball mill. In that case what is understandable that we introduce defect by severe plastic deformation ok. So, we generate a high amount of lattice strain. What where you want to mean we start with a powder and then we mechanically alloy them.

So, that A and B two different type of powder can mix with each other and make a solid solution out of it, so that is the mechanical alloying. And in that particular case, the heavy deformation that is experienced by the powder particle introduce some crystal defect. And once we introduce a defect this means that there is some free energy related with that defect and this defect raise the free energy of the crystalline phase and sometimes the whole free energy may go above of that hypothetical of the amorphous phase.

So, the amorphous phase becomes stable when the free energy of the defect and the crystal and their summation is basically higher than the amorphous phase ok. However, that these G_c that is corresponding to the crystalline lattice and G_d is the free energy due to defect, and G_a is the amorphous phase ok. And if that amorphous phase shows some glass transition we definitely call them as a glassy material, or glassy alloys.

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And so far we have completed the discussion on the glass forming ability and now we need to discuss about the phase transformation in the glassy alloys. The phase transformation means we have discuss about the TTT diagram, and how a TTT diagram looks like. So, in a TTT diagram we think about a temperature and time and here this is the crystal and here this is the T liquid.

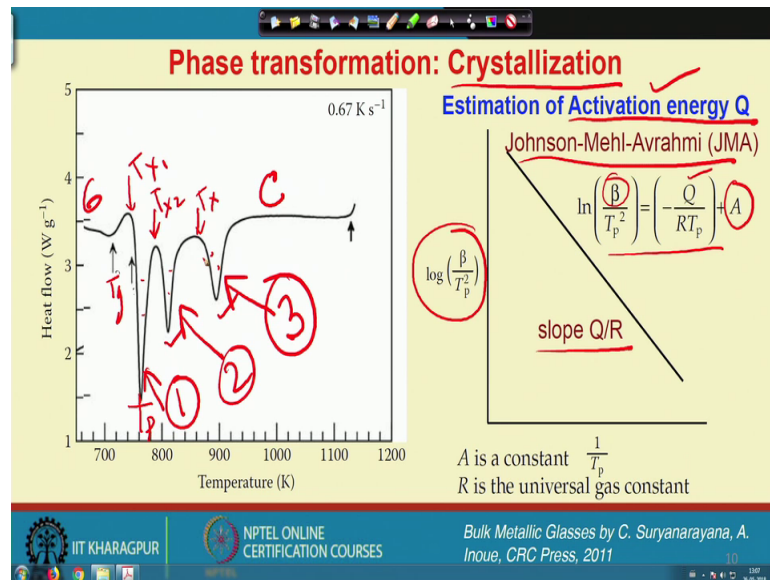
And we basically try to bypass the crystallization event and if T_g is sufficient we can form a glass. However, we can again increase the temperature we will definitely go through a T_g event and then there is a T_x ok. So, these glass to crystal these transformation is also a phase transformation and we are talking about that crystallization event, or it may be also that during cooling itself the liquid transform into two different liquid that is also possible yes.

So, a in a in a glass forming melt or glass forming alloy this kind of phase transformation is possible. When we think about crystallization a crystallization means the crystallization may occur in a bulk volume, or the crystallization may occur in a surface ok. And definitely when it occurs inside a bulk volume we can think about a heterogeneous nucleation, or a homogeneous nucleation ok.

However during cooling itself if there is at all any liquid liquid phase separation then we can think about a spinodal type of decomposition which may be linked with a homogeneous, or may be when a glass transform in to crystal then that glass may

separate out into two different glassy phase. And this is also a phase separation before formation of a crystalline phase. So, these are the possibilities what we can think about in a phase transformation in a glassy alloys.

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Now let us have a look at if there is a crystallization what we can evaluate out of a crystallization event. So, this is a glass in a thermo gram of a glass, and you see here a T_g and here is the T_x. Now, interestingly here you can see two another peaks, which basically means that this is T_{x1}., this is T_{x2}., and this is T_{x3}..

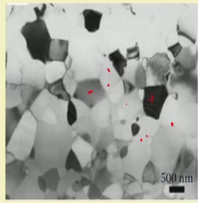
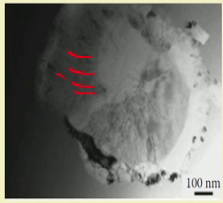
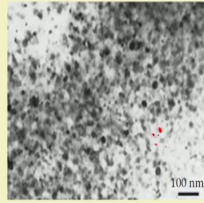
So, there are 3 different crystallization event goes on inside a glass. So, it is not mandatory to have only 1 ok. So, there may be 3 different event goes on. However, we can measure the peak temperature of the event, and we can use the Johnson-Mehl-Avrami equation in order to find out the activation energy required for crystallization. So, this is a mathematically measurements and using DSC, we simply put basically this $\frac{\ln\beta}{T_p}$; here beta is basically let us say the heating rate and Q is the activation energy which we can get from the slope of the curve.



So, from here this is a constant a Johnson-Mehl-Avrami constant A is equal to constant. And we can directly say what is the activation energy of crystallization 1, what is the activation energy of crystallization event 2 or let us say what is the activation energy of

crystallization process 3. So, here was a glass, and here is the crystal where it could be a several crystallization event. And we will be discussing these issues in the next slides.

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Phase transformation: Crystallization

<p>✓ Polymorphous Crystallization</p> <p>Glass-> (No composition change) -> <u>Single Crystalline phase</u></p> 	<p>✓ Eutectic Crystallization</p> <p>Glass-> (large driving force) -> <u>Two (2) Crystalline phase</u></p> 	<p>✓ Primary Crystallization</p> <p>Glass (1) -> <u>Crystalline phase + Glass (2)</u></p> 
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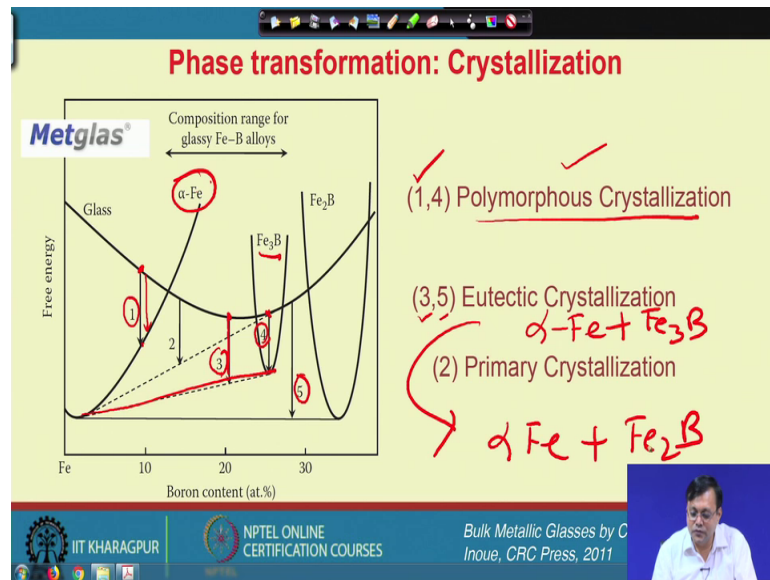
So, overall this crystallization process can be classified into 3 different types. So, people have observed that if we start with a glass then the glass may transform into a single crystalline phase. Now, think about that I have a glass and I have a single crystalline phase. So, there is no need of the change of the chemistry of the glass because the same composition evolve in the in the crystalline phase.

Now, if the glass transform into two different crystal simultaneously together, then this 2 crystal whatever has been appeared inside the glass during heating. So, there must be a very very large driving force and you can see the parallel growth of those crystals. And here this is the only one type of crystalline phase is present ok. Even though there are some dark and these are due to the contrast and orientation contrast; however, there could be a primary crystallization. So, these are the 3 major types.

So, in case of primary crystallization a glass basically changes it is composition to glass 2 and another crystalline phase will appear, so this is the primary crystallization. What it interesting here you must look at that when a glass 2 appear then definitely there is a need of the change in the chemistry of the glassy phase and there must be a phase to be separated ok.

So, this is a primary crystallization along with a phase separation. So, here this is the glassy phase along with some crystalline phase. And we can think about the thermodynamics of such events and we can take an example which the very very common Metglas.

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So, metglas is iron boron alloy and here we have shown you the free energy curve of the glassy phase ok. And now let us have a look at that if I take case 1, so I start with a glass from a glass it simply without any change in the chemistry of the glassy phase I get a crystalline phase which is alpha iron. And so it is a polymorphous crystallization this is case 1. Whereas, I can also may have where alpha iron is basically solid solution that is why there is a continuation of this free energy curve.

And now if I have created a simply inter metallic phase then in case of inter metallic you see this is case 4, where a glassy phase transform into a Fe_3B . So, this is also a polymorphous crystallization. However, in case of 3 and 5 so in case of 3 and 5, so if I start with such a composition and then a crystallize it then I will have a chance of getting alpha iron, alpha iron plus Fe_3B ok. So, this is a in case 3 or maybe I can also have that that alpha iron, or I will have Fe_2B ok. So, these are the eutectic crystallization. So, both the phase will appear in case of 5 ok.

And now the primary crystallization will appear in case of 2, because I start with this composition and the same glass will have a different composition right. So, here when I

there is a decrease in the free energy, so I will have one phase which is alpha iron and I will create a glass which has a different composition than the starting glass and this is called as a primary crystallization.

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Annealing (sub T_g): Structural Relaxation

Changes in the Bulk Densities, ρ (g cm^{-3}) of Metallic Glassy Alloys in the As-Solidified and Structurally Relaxed Conditions

Alloy Composition	Synthesis Method	Rod Diameter (mm)/Ribbon Thickness	Density (ρ)		$\Delta\rho_{\text{relaxed}}$ (%)
			$\rho_{\text{as-solidified}}$	ρ_{relaxed}	
Pd _{77.5} Cu ₆ Si _{16.5}	Melt spinning	30 μm thick ribbon	10.46	10.51	0.48
Pd ₄₀ Cu ₃₀ Ni ₁₀ P ₂₀	Melt spinning	40 μm thick ribbon	9.318	9.337	0.2
Pd _{77.5} Cu ₆ Si _{16.5}	Water quenching	2 mm dia rod	10.48	10.51	0.29
Pd ₄₀ Cu ₃₀ Ni ₁₀ P ₂₀	Cu-mold casting	5 mm dia rod	9.27	9.28	0.11
Zr ₅₅ Cu ₃₀ Al ₁₀ Ni ₅	Cu-mold casting	5 mm dia rod	6.82	6.83	0.15

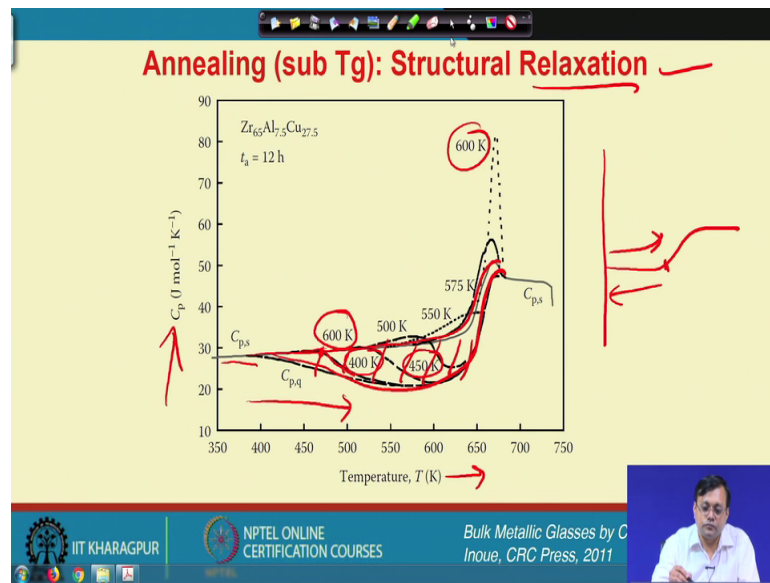
Note: $\Delta\rho_{\text{relaxed}} = \frac{\rho_{\text{relaxed}} - \rho_{\text{as-solidified}}}{\rho_{\text{as-solidified}}}$

Now I may also have a annealing event which is the structural illustration and in that particular case a sub T_g annealing is required. A sub T_g annealing gives us that how much relax volume we have created.

So, a relax glass always have a higher density than the as solidified glass there are example of such and we can see the density difference. By relaxation phenomena, means if we heat up a glass then inside the glass the excess free volume will release. So, this is called as a structural relaxation.

So, in that particular process we can also have a chance of increasing the density of the glass. So, this is the density of the relax glass and this is the density of the as solidified glass and you can see as we increase the relaxation the density is also increasing.

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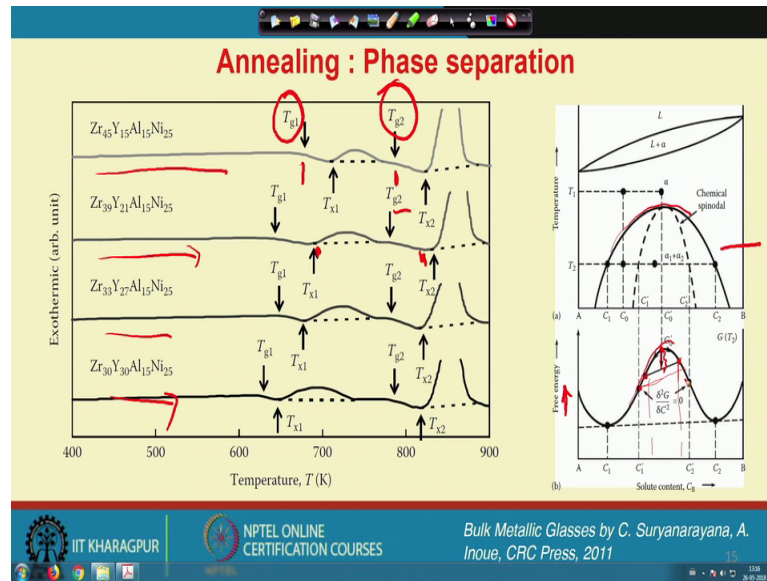


Now, I have magnified here the glass transition event of a thermo gram of a glass transition event. And so here this is the C_p in the y axis and here is the temperature.

So, from here, you see if I pre anneal a glass let us say 600 K, or let us say 450 K, or 400 K. So, after annealing if I again re heat so, let us say the initial plot was like that and I have annealed it close to T_g , I cool it down again I reheat the same. Then initially the glass shows such a glass transition, but after annealing the relaxation after annealing means after relaxation it follows this line.

So, this is the enthalpy which is related with the relaxation which is exothermic in nature. And therefore, we can see that if a relax glass and an as annealed glass we will not follow the same path. So, a relaxed glass will follow this path than a annealed glass. So, how much we have relaxed a glassy structure can be visualized from the C_p curve.

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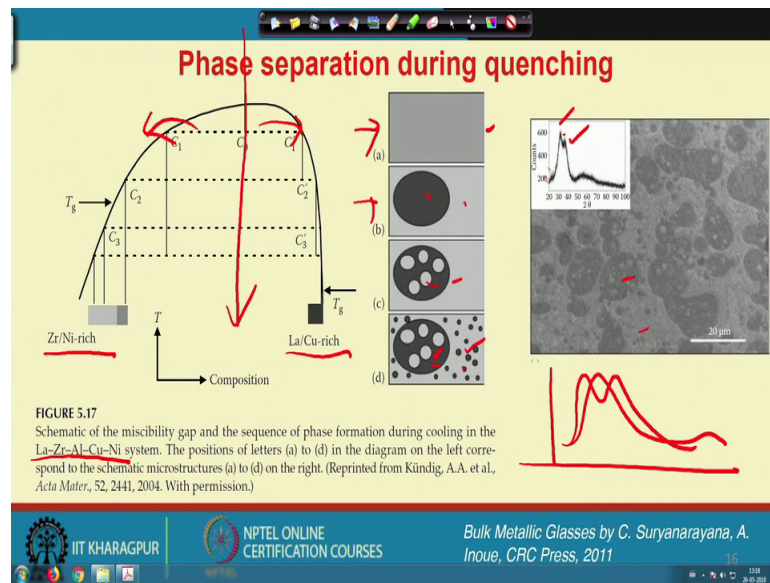
Now, this is case of a phase separation I have shown you the plots of different thermograms. So, these are for different alloys and you can see that it is also possible to see two different glass transitions in a same glass. So, a two different glass transition simplifies the there must be two different glassy phase.

So, this is one glass transition temperature, this is another glass transition temperature. But even though the crystallization started here, but here there is a second crystallization event followed by another glass transition. So, this is another question come to the mind that whether this kind of glasses how they have appeared in the liquid.

So, one can think about the classical way of understanding a phase separation in a solid. In a solid solution, here there is a spinodal and if you look at the free energy curve then definitely here if it is the composition of the average composition and then there is always a chance that there is a decrease of the free energy and we can get two different region with two different competition ok. And this is an automatic process by which we can go to the to the two different composition ok.

So, I can also have a glass where the glass if we provide some extra thermal energy, then the glass will dissociate into two different compositions. So, in a solid state itself the glass can be there will be a phase separation. So, this is one of the ideas that came because of such kind of even that people find out. Now, the phase separation can also happen in the under cooled liquid during solidification itself.

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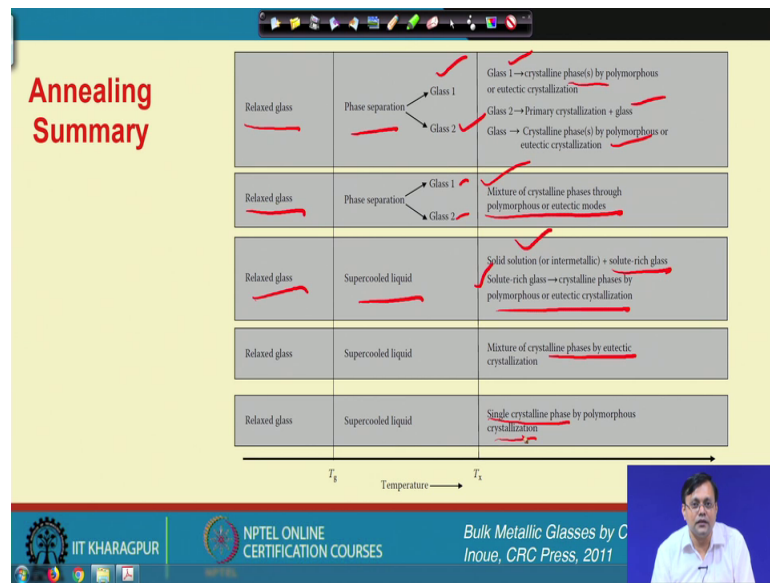


So, let us say I may have created a in a in a under cooling liquid a zirconium rich, or let us say a lanthanum this is a lanthanum, zirconium, aluminum, copper, nickel system, we are talking about. So during cooling itself the liquid phase separates into two different region which is schematically shown here. So, this was the initial liquid and then there are some other region appears and then two different regions appear two different chemistry ok. And then ultimately this 2 regions solidified into a into a single glass ok.

And so definitely I will have two different phase and two different phase means two different glass transition temperature. So, overall the micro structure if you look at the solidified micro structure has this kind of phenomena ok. And if we do X-ray diffraction in normal conventional x diffraction of a glass show you a single hump ok.

And in case of such kind of 2 phase phase glasses instead of a single hump you will see 2 hump which is appear here. So, two different hump is corresponding to a two different phase in the amorphous phase region.

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So, these things makes a more relevance to the subject and we try to summarize what we have discussed so far. So, in case of a relax glass we can think about a phase separation into glass 1 and glass 2 where this glass one may undergo some sort of crystallization. Let us say primary crystallization, or let us say polymorphous or eutectic crystallization or may be glass 2 also go for such kind of phenomena.

Maybe we can again start with such a relax glass where there are two glasses where we get a mixture of crystallization phases through polymorphous or eutectic mode. However, there may be super cool liquid and we may produce a solid solution, or inter metallic that I have shown you in case of a Fe-B- Metglas, and we may have created a solute rich phases.

So, this solute rich glass may undergo some sort of polymorphous or eutectic crystallization or maybe we can go for a mixture of crystalline phases plus eutectic. So, these are so many different possibilities like a single crystalline phase by polymorphous or crystallization. So, when we think about a crystallization or a phase transformation in a glass not only crystallization is important, but also phase separation is important. So, here we are talking about phase separation means difference in their chemical short range order.

Thank you very much we will discuss the other properties of metallic glasses in the next classes.