

Non - Metallic Materials
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Module – 03

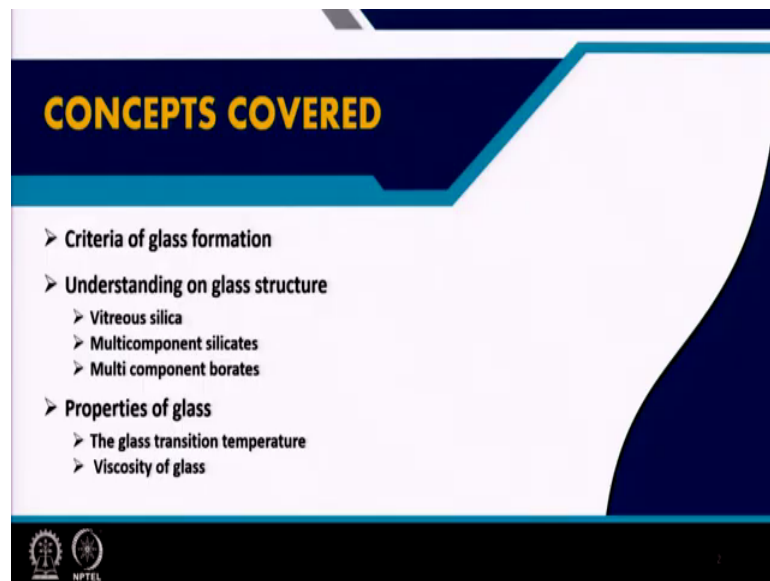
**Diffusion, phase transformation in non – metallic materials, glass and glass -
ceramics**

Lecture – 16

Understanding on conventional glass and amorphous solids

Welcome to my course Non-Metallic Materials. And today, in this part of module 3, Diffusion phase transformation in non-Metallic materials, glass and glass ceramics. This is lecture number 16, where I will be taking you through Understanding of conventional glass and related amorphous Solids.

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Criteria of glass formation that will be elucidated in this lecture. In the last lecture, we talked about the nucleation and growth of crystalline phase in a melt which is not required for a glassy substance. So, we talked about critical cooling rate, the rate should be maintained, so that the glass will form and it does not crystallize, so that was explained.

And we will understand the structure of the glass particularly the vitreous silica, several multi component silicates as well as multi component borate composition. Then,


properties of the glass to very important property in the terms of glass transition temperature and the viscosity of the glass will be introduced.

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Criteria for glass formation

1. Low nucleation rate. ΔS_f is small; crystal/liquid interfacial energy γ_{cl} is high. For both of this ΔG_c is large and nucleation is difficult.
2. High viscosity at or near melting point, η_m . This translates to low growth rate
3. Based on the above, $\Delta S_f / \eta_m$ ratio should be small.
4. Absence of heterogeneities. Heterogeneities can act as potent nucleating agents and reduce the critical nuclei size and thus, enhance the nucleation kinetics.

Compound	Melting point °C	ΔS_f J/(mol·K)	η_m Pa·s	$\Delta S_f \times 10^4 / \eta_m$	Comments
B ₂ O ₃	450	33.2	5000	0.0066	Excellent glass former
SiO ₂	1423	4.6	2.3×10^3	2.0×10^{-3}	Excellent glass former
Na ₂ Si ₂ O ₇	874	31.0	200	0.155	Good glass former
Na ₂ SiO ₃	1088	38.5	20	1.9	Poor glass former
GeO ₂	1116	10.8	71.428	2×10^{-4}	Excellent glass former
P ₂ O ₅	569				Glass former
NaAlSi ₃ O ₈			3.2×10^3		Glass former
CaSiO ₃	1544	31.0	1	31.0	Difficult to form glass
NaCl	800	25.9	2×10^{-1}	1.3×10^4	Not a glass former



So, again I will go back to the concept of my last lecture. The criteria for glass formation is that the nucleation rate should be low. So, for that case the entropy of formation should be small and crystal liquid interfacial energy that should be high. So, for both this if it is there the barrier is quite large, so the crystallization is very difficult. So, I think you understood from my last lecture that how exactly it happens, how to control the crystallization part.

So, another important thing is that while in the melt, the viscosity of the melt should be very high. So, if the viscosity is very high then the growth of the crystalline part, once your cooling the crystal growth will be minimized, because the atomic diffusion will be retard it, so the glass formation will be expedite.

So, that basically tells you that both the entropy term and the viscosity term. This in case of the glass formation the entropy should be small and the viscosity should be high. So, this ratio of the entropy and viscosity they should be small.

And also there should be absence of heterogeneity because the heterogeneity acts as a nucleating agent and that reduce the critical nuclei size and therefore, enhance the nucleation rate. So, heterogeneity should be avoided.

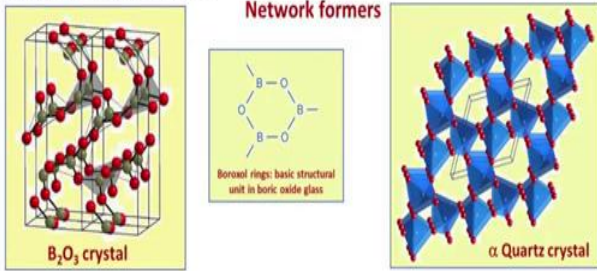
So, if you want to maintain all these criteria there are very handful of materials that I have listed here, that boron oxide, silica, sodium silicate, germanium oxide, you see the melting point, you see the value of the entropy, you see the viscosity and you see this ratio. So, progressively you see that most of them they are having very low values. So, all of this material they are excellent glass former.

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Structure of glass

If cooled rapidly enough (splat cooling), any liquid will form a glass: Glasses have indeed been formed from ionic, organic, and even metallic melts.

Network formers



The diagram illustrates the structural differences between crystalline and amorphous forms of boron oxide. On the left, a 3D ball-and-stick model of a B_2O_3 crystal is shown within a wireframe unit cell, with red spheres representing oxygen atoms and grey spheres representing boron atoms. In the center, a 2D chemical structure shows a six-membered boron ring (B₃O₃) with the caption: "Boron rings: basic structural unit in boric oxide glass". On the right, a 3D ball-and-stick model of an α Quartz crystal is shown, consisting of a continuous network of silicon (grey) and oxygen (red) atoms. A small inset photo of a man in a suit is visible in the bottom right corner of the slide.

B_2O_3 crystal

α Quartz crystal

The glass forming oxides having continuous three dimensional network linked polyhedral and are known as network formers. B_2O_3 , SiO_2 , P_2O_5 , GeO_2 are examples

So, if you consider the structure of the glass. Particularly, if you consider the alpha quartz structure and B_2O_3 kind of crystal structure, so they are called network former. So, they form a linkage structure in their crystalline state, but when they are in molten state, then the viscosity is so high that once you cool it down they never go back to their crystalline phase. So, they always form the glass. So, this P_2O_5 boron oxide silicon dioxide they are termed as a glass former.

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Vitreous silica

Long range order: crystalline quartz

Short range order: vitreous silica

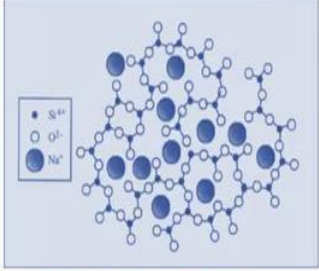
- Basic building block of all crystalline silicates is the SiO_4 tetrahedron.
- Every silica tetrahedron is attached to four other tetrahedra and a three dimensional periodic network.
- For **fused silica** the network has no long range periodicity

Now, the difference between the crystalline structure and the glassy structure is apparent in this view graph. In crystalline structure, you see that there is a long range ordering if you see that this kind of coordinated building block tetrahedra of silica if it is there if you go to larger distance then this crystalline this long range ordering is maintained of this SiO_4 tetrahedra.

So, each of this silica tetrahedra is attach to 4 other tetrahedra 3 in plane one upward and it produces a 3 dimensional network. Now, once it is fused you melt it at very high temperature and then cool it down the viscosity is so high, then this regularity is not maintained.

So, it is having a very long very short range order unlike the long range ordering in crystalline structure. So, it form a glassy structure. So, this glassy structure is a low packed structure. It is not favorable intense and there is no long range order, it is short range ordering that you have in this material.

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Multi – component borates

In pure B_2O_3 each B is bonded to three O atoms and each O to two B atoms. Borosilicate glasses are common where B_2O_3 and SiO_2 are mixed with alkali and alkali earth metal oxides. Beyond 0.2 M fraction of the modifier used NBO starts to appear. At lower fraction only BO_4 tetrahedral forms.

Multi – component silicates

- Alkali or alkaline earth oxides added to silica melt create **non – bridging oxygen (NBO)**. These oxides are referred to as **network modifier**.
- As shown, continuous network structure is broken up due to the presence of NBO.
- Al_2O_3 , when added to silica glass melt might act as network former or network modifier.
- 0 – 0.1 M M_2O acts as network modifier, however, for 0.1 – 0.33 M M_2O , discrete $(Si_6O_{13})^{6-}$ ring exists



Now, pure silica it is having a very high melting point. So, it is very difficult for you to maintain the melting temperature that high. So, you can reduce the melting temperature by adding several modifier into the structure. So, usually the modifier are alkali or alkaline earth oxide that is added to this silica melt.

And what it what it does, it just occupy the position in between this silica tetrahedra and in the process it breaks some of the bonds and as a result in the silicate structure some oxygen they are having a dangling kind of bond. So, this bond is broken.

So, otherwise each of this oxygen is connected with at least two silica ions and you see this type of oxygen they are broken. So, this type of oxygen is termed as non-bridging oxygen. So, there are several non-bridging oxygen that is introduced in the structure by the addition of this ah modifier which is alkaline oxide.

So, the continuous network structure is broken now, and the addition of or the appearance of this non-bridging oxide that is there. So, certain oxide it is difficult for you to understand whether it acts as a network former or it is a modifier. So, as a network former it goes into the structure. So, alumina is one of the examples.

So, if you add very small concentration of alumina in silica melt, it acts as a network modifier, typically 0.1 mole fraction if you use it, then it acts as a network sorry; it acts as a in in in a typical concentration it acts as a network former and otherwise it acts as a network modifier, when it creates the non-bridging oxide.

So, usually for certain other oxides it can act as a network modifier at lower concentration, but when you add at higher concentration level, then instead of this kind of structure it forms a discrete rings of silicate that is also possible in certain other modifier you know crystals modifier oxides.


So, multi-component borate is also possible. So, you can add two glass former like boron oxide and silicon dioxide mixed in different proportion. So, once the proportion of B_2O_3 is very small, then it does not form any noticeable change into the structure the structure is still rigid. But once a certain content if you exceed it acts like a modifier by the creation of the non-bridging oxygen into the structure.

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Some important commercial glass composition

	Network formers			Network modifiers					Softening temp. °C
	SiO ₂	B ₂ O ₃	Al ₂ O ₃	Na ₂ O	K ₂ O	MgO	CaO	PbO	
Fused silica	99.8					0.1	0.1		1600
Vycor	96.0	3	1						
Pyrex	81.0	13	2	3.5	0.5				830
Soda silica	72.0		1	20.0		3.0	4.0		
Lead silica	63.0		1	8.0	6.0		1.0	21	
Window	72.0	1	2	15.0	1.0	4.0	5.0		700
E glass	55.0	7	15	1.0	1.0		21.0		830

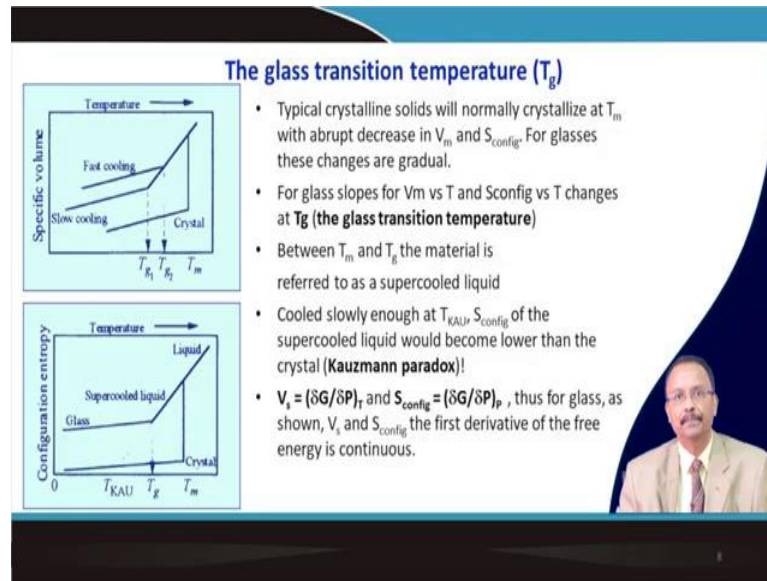
Once glass forms, unlike crystalline solids, the change is not from disorder to order, but rather from disorder to disorder with less empty space.



So, some of the typical compositions of commercial glass, fused quartz this is having very large softening point and also very large melting point. And vycor glass is something similar to this although it is much cheaper the vycor glass it is formed in a very special way. So, it contains 96 percent silica. Other than that this pyrex where a substantial amount of B_2O_3 is mixed.

So, it is a borosilicate type of glass. And common soda lime silica glass or soda silica glass, window glass contain the soda lime silica, and its melting point is progressively reduced. So, whenever you use this modifier it breaks the bond of silicate and that introduce the non-bridging oxygen and the melting point is progressively reduced. So, the formation is easier. So, these are commercial glass compositions.

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Now, the property that is important for the glass to form is the glass transition temperature. So, if you consider a crystalline solid and you start to cool it down from its melt then at melting point there is a sudden drop of the specific volume, and then it crystallizes it forms crystal and specific volume change is very dramatic and then once you cool it down, then it reduces the specific volume reduces, but at much lower slope as compared to the melt.

But in case of glass it does not happen like this. So, it continuous this line is continuous. So, it is liquid like structure, then at a characteristic temperature you see that it start to form, this is a liquid like region and then it form a glass and this transition is the glass transition temperature.

So, in between this T_m and this glass transition temperature it is termed as a supercooled liquid. So, if you cool it relatively slow you will find that the glass transition temperature it reduce and in case of fast cooling the glass transition temperature is at relatively higher part, I mean higher temperature regime. So, if you now consider the configurational entropy of this glassy material.

Then, if you cool down at a fairly strong very cooled relatively slow effect then you reach a region here you the same slope is maintained. So, you have a configurational entropy which is actually lower than your crystal. So, this is a paradox, right. So, from the crystal the configurational entropy may not be lower. So, this is known as Kauzmann

paradox that is particularly relevant for the glass material. And this we will come back to this later on.

And the first derivative of the free energy as a function of pressure at constant temperature and with constant pressure at constant temperature if you do the first derivative then it is first derivative of the free energy is continuous. So, if it is continuous then we learnt from our phase transition lecture that this is this could be a second order phase transition.

So, you can do the second derivative of ΔG as a function of temperature, and then you can derive this thing in terms of a thermal expansion and or in terms of a specific heat. So, both are discontinuous as you can see, for this and for this also, both for this specific heat at constant pressure as well as the thermal expansion you get a real break and that is confirming that this one is a second order type of phase transition.

So, T_g is a function of a cooling rate as I have shown that with slower cooling rate they shifts to lower temperature. And so, that tells that T_g is a kinetic quantity it is not a thermodynamic property and this composition also effect T_g . So, T_g basically measure the rigidity of the glass network structure. So, when you are adding a modifier you that tends to reduce the T_g while you add a network former it will try to increase it. So, it is dependent on the composition.


So, for some oxide it really depends that whether that oxide will act as a network former or it will form a non-bridging oxide. So, the measurement of T_g if you do then that in fact, can confirm this thing.

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The glass transition temperature

Measurement of glass transition temperature

- Any of the properties namely, V_v , S_{config} , C_p or α as a function of T (cooling rate) can be measured to determine T_g . The temperature at which the properties *changes slope* whether continuously or abruptly is defined as T_g
- DTA experiment can be performed to measure T_g . For a glass that does not devitrify, T_g is a weak endothermic process (a).
- If the glass devitrify, then exothermic crystallization peak along with endothermic signature due to melting would appear (b).



So, usually the T_g you can measure by a technique called differential thermal analysis, where as you have seen that in case of a glassy structure if you heat it then at certain point there is a slight change in the slope before it goes to the under cooled state.

And this just changes its slope and in case of a system, where it crystallize you will see that this slope change is there and in between melting you get in fact, up to the melting point you get a exothermic peak, which denotes the crystallization and then followed by a melting, where it is done by this indicated by this endothermic peak.

So, both this comparing these thing you can understand whether it is a basically a glassy structure or it will the glass will devitrify and accordingly you can measure the glass transition temperature.

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Viscosity of glass

Viscosity determine several important temperatures correspond to **melting, working, softening, annealing** and **strain points** used in glass industries.

Viscosity (η) = $F d / (A v)$; ratio of applied shear stress : rate of flow v of a liquid. The liquid is assumed to be confined between two parallel plates of area A and distance d . Shear force F is applied

Thus, $\eta = \tau / \dot{\epsilon}$ (shear stress / strain rate) \sim Pa.s.

It changes in more gradual fashion with temperature.

Strain point - $\eta = 10^{15.5}$ Pa.s (Lehr furnace operation)

Annealing point - $\eta = 10^{14}$ Pa.s

Softening point $\eta = 10^{8.6}$ Pa.s

Working point $\eta = 10^5$ Pa.s (readily shaped)

So, the viscosity of the glass apart from the transition glass transition temperature is another important thing. And viscosity actually determine several important temperature characteristic temperature, which is very important for the glass industries. So, viscosity correspond to the melting working temperature softening temperature of the glass annealing temperature strain point they are very well defined.

So, as you know the viscosity is a ratio eventually it is ratio of a shear stress by strain rate and the unit is Pascal second. So, shear stress that is Pascal and the strain rate, this is per second because strain and then strain rate is per second, so it is Pascal second.

So, you can measure the viscosity I will explain how the glass viscosity is measured and you can define the strain point where the viscosity of the glass is a very specific 10 to the power 15.5 Pascal second. So, in the strain point you heat it in a layer furnace. So, all the defects etcetera is annihilated.

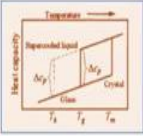
Annealing point is 10 to the power 14 Pascal second. So, it is here and then it is a softening point at that little bit higher temperature and the composition depending on the composition this temperature changes, but the viscosity remain same. So, the softening point is 10 to the power 8.5 or 8.6 Pascal second and working point is 10 to the power 5 Pascal second.

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Viscosity of glass

Understanding on the temperature dependence of viscosity


The functional dependency can be fitted by Vogel – Fulcher – Tammann (V – F – T) equation $\ln \eta = A + B/(T - T_0)$, A, B, and T_0 are three temperature independent fitting parameters.



Liquid is divided into N_c blocks, each containing n atoms where $n = N/N_c$, N is the total number of atoms in the system. For each block only two configuration exists. Thus, in each block from existing only one type of new configuration would be possible. Configurational entropy in each block is $k \ln 2$, thus, total configurational entropy of the supercooled liquid $\Delta S_{\text{config}} = N_c \cdot k \ln 2$

$\Delta S_{\text{config}} = N/n \cdot k \ln 2$
 $n = N/\Delta S_{\text{config}} \cdot k \ln 2$. **Now look at the c_p vs T plot presented earlier**
 Δc_p at T_g is equal to its value at T_k
 $\Delta S_{\text{config}} = \Delta S_{\text{config}}^{\text{SCL}} - \Delta S_{\text{config}}^{\text{glass}} = \Delta c_p \ln T/T_k$

$n = N \cdot k / (\Delta c_p \ln T/T_k) \cdot \ln 2$



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So, these are the characteristics viscosity as a function of temperature that is very important to understand the glass structure, and composition and viscosity they are related to this type of typical plot.

So, this temperature dependence of the viscosity that is can be fitted by this type of relation, where A constant plus another constant B and this is the temperature and this is a fitting parameter. In fact, this A, B and this T_0 , they are all fitting parameter and that very well can define the kind of viscosity temperature diagram, which I just talked about. So, this is known as Vogel Fulcher Tammann equation and abbreviated as V-F-T and let us try to understand that what exactly is happening.

So, once you are cooling the liquid from high temperature, so for example, you divide it at the N_c number of block within this liquid matrix and each of this block that contain N number of atoms. So, number of atom is your basically N divided by N_c , when N is the total number of atom in the system.

So, for each block there are only two configuration exist and. So, you can always think in terms of a configurational entropy term in case of the super-cooled liquid and already I derived the configurational entropy is only two types of variation is possible, so it is $K \ln w$, so here w is 2. So, you put it back to this relation of number of atoms and you can derive this number of atom and relate it with the configurational entropy.

Now, you look at this curve in the super-cooled liquid region how this, how this specific heat at constant pressure changes. So, there is a drop here, and in case of ah the super-cooled liquid this can be extended, but this separation remains almost same.

So, here I can relate this configurational entropy of this glassy matter with the specific heat of the super-cooled liquid by this relation and then, that allows me to calculate the number of atom in this block whatever I referred to with this relation the total number of atom is there, change of the specific heat capacity is there.

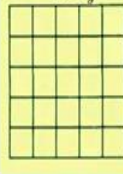
And one characteristic temperature T_k that I have defined here which does not have any physical meaning, but the number of atom in those hypothetical block that you can determine by this relation.

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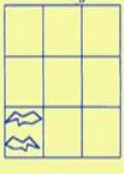
Viscosity of glass

$$n = N.k/(\Delta c_p \ln T/T_k) \cdot \ln 2$$


$T > T_g$



$T \approx T_g$




$T = T_k$



As the temperature is lowered and various configurations are frozen out, the cooperatively rearranging regions decreases in number and increases in volume. At T_k only one configuration remains and there is a total loss of configurational entropy

Configurational entropy is related to the rate of molecular transport. As the blocks become larger, it takes more time for them to switch configurations. Hence relaxation time τ is proportional to n

$$\tau = (\text{constant}) \cdot \exp[\xi \cdot n]$$


Now, if you change the temperature now, then you see that at relatively high temperature you have this number of block, this is quite large and then progressively as you cool it down then this number is this this block number these are relatively less as compared to high temperature.

And at that characteristic temperature which is T_k , I defined that here it is completely disappeared. So, what is happening at the temperature you lower down at various configuration they are frozen out, and the region in between they are rearranging the regions in number and increase in the volume.

So, here the volume is increasing and number of this regions are progressively reduced and there are two different configuration in each of this block and then finally, at the temperature a characteristics temperature there is a total loss of configurational entropy.

So, the configurational entropy is related to the molecular transport and as the block you can see it is becoming larger, it takes more time for them to switch from one particular configuration to other inside it. So, the relaxation time that is related with this number of atom by this simple relation a constant term is involved and exponential zeta into n number of atom that is there.

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Viscosity of glass

$$n = N.k / (\Delta c_p \ln T/T_g) \cdot \ln 2$$

$$\tau = (\text{constant}) \cdot \exp [\xi \cdot n]$$

Relaxation time τ (characteristic time for structural relaxation) \sim viscosity (η); ξ is a constant
 $\eta = K^A \cdot \exp [\xi \cdot n]$ substituting the value of n into this expression
 $\ln \eta = \ln K^A + (\xi N.k \cdot \ln 2) / (\Delta c_p \ln (T/T_g))$

$$\ln \eta = A' + B' / \ln (T/T_g)$$

Compare this with V-F-T relation $\ln \eta = A + B/(T - T_0)$. Put $T_0 \approx T_g$ and compared with the derived one as assignment.

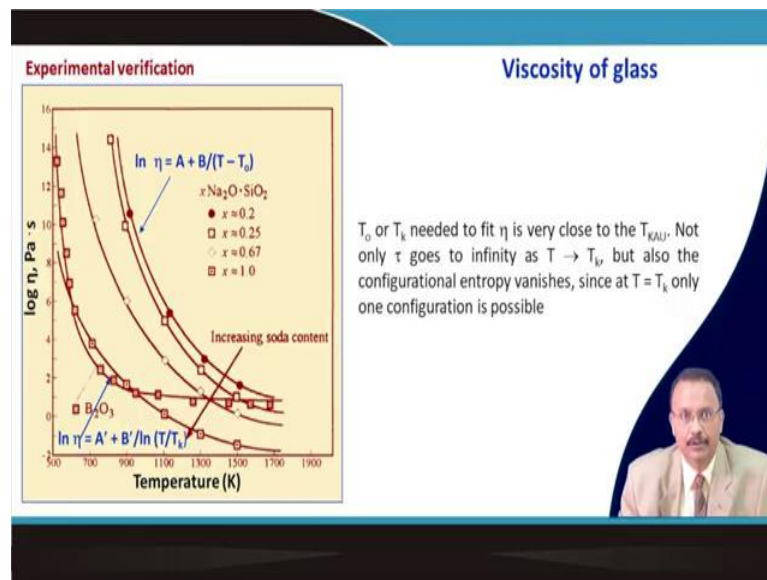
So, I can define two equation, 1 is the number of atoms in those hypothetical block and another one is their relaxation time, characteristics relaxation time. So, which is also having this n component. So, now, you can replace this n with this value and then you can do little bit of algebra and eventually this tau is some way related to the viscosity of the solution.

So, the relaxation time, this is a characteristics time for this kind of relaxation to take place. It is almost equivalent to the viscosity because lowering the temperature it is basically frozen. So, that viscosity can be replaced intermittently with this time, because it is Pascal second relaxation is also in second, so you can replace this.

And then you come up with this relation, which is very similar to the Vogel Fulcher Tammann relation that I talked about. Not exactly identical, but this was A plus B divided by T minus T₀, where T₀ is a characteristics temperature. And here you are finding ln of the viscosity is A plus B divided by ln of T by T_k.

So, not exactly similar, but it is comparable when you take this characteristic T₀ temperature with T_k, so in one of the assignment problem it is given to understand this similarity.

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And the actual data of several glass composition with different amount of this sodium oxide that has been taken into consideration. So, all this solid lines are fitted along with this V, F, T relation. And only the bismuth base composition is fitted with the relation that we have just derived. So, both these are quite close to each other.

So, this tells us that the configurational entropy that actually vanishes at that characteristics temperature, which was defined by T_k in our curve involving the specific heat versus the temperature that we just talked about.

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Effect of glass composition on viscosity

Legend:
 □ Li₂O @ 1400°C
 ○ K₂O @ 1600°C
 ● BaO @ 1700°C

Viscosity of glass

- It is difficult to break high-energy directional Si-O-Si bonds. The viscosity of pure liquid SiO₂ at 1940 °C is 1.5 x 10¹⁴ Pa.s
- Addition of basic oxide like CaO and Na₂O makes NBO and break down the 3d network into progressively smaller discrete units.
- The effect of the addition in reducing the original viscosity is dramatic (see the Figure)

So, the glass composition effects is viscosity for example, when silicon dioxide is considered, it melts at very high temperature and the viscosity at the melting point is quite high. But basic oxide when you add lithium oxide or potassium oxide or barium oxide then progressively the viscosity drops down. So, it is easier for you to work with this glass composition.

And it is indeed a very dramatic effect the way you can reduce the viscosity at a particular temperature you see the kind of difference that you get at this particular temperature, if you want to have reduce the viscosity it dramatically change with the addition of the loop and content.

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Measurement of the viscosity

L is the length of concentric cylinder
 T torque is measured on the stationary outer cylinder
 The inner cylinder is rotated at an angular velocity ω_a
Up to 10⁷ Pa.s can be measured

$$\eta = \frac{(b^2 - a^2)T}{4\pi a^2 b^2 L \omega_a}$$

Viscometer working principle

Viscosity of glass

$$\eta = \frac{mg}{3\pi R^2 \dot{\epsilon}} = \frac{mgL_0 t}{3\pi R^2 \Delta L}$$

Elongation ΔL at time t is measured applying a known load (mg) at the end of fiber

Now, viscosity is actually measured by two different way, when the viscosity is up to 10 to the power 7 Pascal second, then you use a concentric cylinder and between this you add this glass melt is added here, and the inner one it is rotated and outer one remains fixed against a spring. So, that you can calculate the torque. You know the length of the cylinder, you know the angular speed and you know the dimension of the cylinder, so you put it back in this relation and viscosity you will get.

If you want to measure the viscosity at relatively higher level, when the viscosity is high then you use in the form of a fiber in a cantilever position. So, you know the length of the fiber, you know the diameter of it, and you know the known weight until it expands.

So, you measure the change in length, you know the weight, you know the radius of the curvature, and you know the time involved where this strain straining has taken place. So, with that you can measure the viscosity when it is more than 10 to the power 7 level.

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So, this is the reference from the book by Barsoum and several other good standard textbook that is referred.

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CONCLUSION

- Introduction of glass
- Concept of homogeneous and heterogeneous nucleation
- Crystal growth : Standard growth model, Surface nucleation growth, and screw dislocation growth
- Kinetics of glass formation, estimation of critical cooling rate and its significance
- **Criteria of glass formation**
- **Structure of glass**
- **Glass transition temperature and viscosity of glass: Basic understanding**
- **Measurement of glass viscosity**

NPTEL

And in this lecture along with the earlier lecture part, the first lecture as well as in this lecture we have considered the introduce the concept of the glass, homogenous and heterogeneous nucleation, crystal growth, standard growth model, surface nucleation growth and screw dislocation growth.

Then, we talked about the kinetics of the glass formation, estimation of critical cooling rate, and then criteria of the glass formation was defined, structure of the glass was introduced, glass transition temperature as well as the viscosity the basic understanding of these two important parameters are defined and finally, measurement of viscosity has been described.

Thank you so much for your attention.