

**Non - Metallic Materials**  
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**Module - 07**

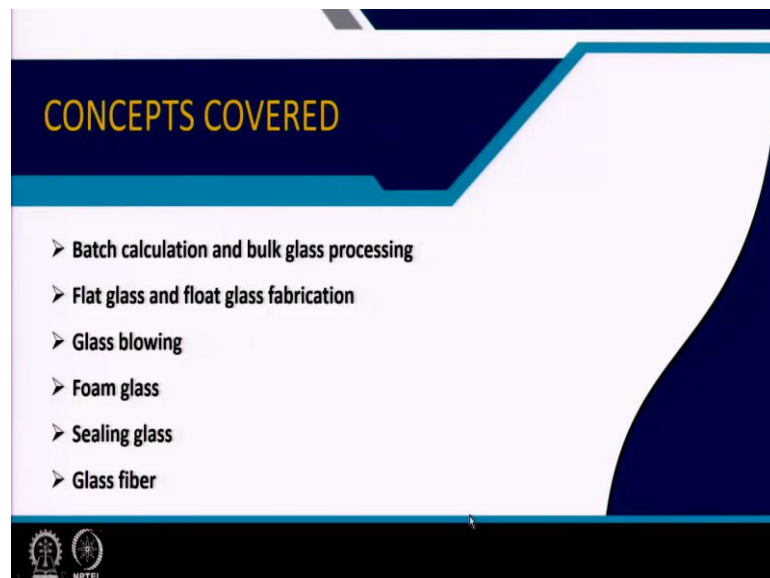
**Processing of non - metallic materials, sintering and microstructure development**

**Lecture - 39**

**Processing of glass and amorphous non - crystalline solids**

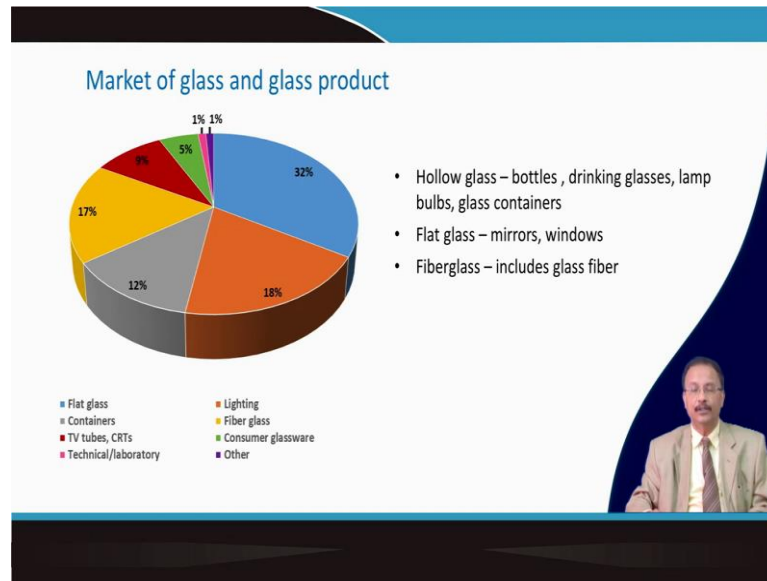
Welcome to my course Non-Metallic Materials and we are in module number 7 Processing of non metallic materials, Sintering and microstructure development. We are in lecture number 39 Processing of glass and amorphous non-crystalline solids.

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So, in this particular lecture, we talk about batch calculation which is so important for glass manufacturing then, we will be talking about flat glass and float glass fabrication process, the flat glass fabrication process is almost obsolete nowadays. Then, we will discuss about the glass blowing, then how the foam glass is made that is actually used for thermal insulation then, sealing glass and glass fiber fabrication will be described.

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So, if you see the market of the glass product that mostly you can see different types of glasses like fiber glass, then this TV and CRT tube nowadays it is becoming less and less relevant, but it was sizable a decade back then you have containers then various types of light cover, then flat glass is of course about 32 percent is flat glass in mirror and windows as a building material.

Glass fiber is a good reinforcing agent so, that is also having a sizable proportion and bulk use. And actually we need to know that how these different articles like hollow glass article bottle, drinking glasses, lamp bulbs, glass container etcetera they are used and also this product like window glasses how exactly they are made.


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**Concept of batch calculation: Soda – lime – silicate glass** Processing bulk glasses

- Soda – sodium oxide ( $\text{Na}_2\text{O}$ ); lime – calcium oxide ( $\text{CaO}$ ), silica – silicon di-oxide ( $\text{SiO}_2$ )
- All manufacturers of say flat glass **use same formula**, but never actually use the compounds  $\text{Na}_2\text{O}$  or  $\text{CaO}$ .  
As you remember composition :72 wt%  $\text{SiO}_2$ , 14 wt%  $\text{Na}_2\text{O}$ , 9 wt%  $\text{CaO}$ , 4 wt%  $\text{MgO}$ , 1 wt%  $\text{Al}_2\text{O}_3$  (see the following Table, how the molecular formula has been calculated)

Oxide	Wt (g)	Molecular weight (g/mol)	Wt%/molecular weight (mol)	Ratio (divide each by smallest ratio)
$\text{SiO}_2$	72	60.1	1.20	120
$\text{Na}_2\text{O}$	14	62	0.23	23
$\text{CaO}$	9	56.1	0.16	16
$\text{MgO}$	4	40.3	0.10	10
$\text{Al}_2\text{O}_3$	1	102.1	0.01	1

**Molecular formula** :  $\text{Al}_2\text{O}_3$  10  $\text{MgO}$ . 16  $\text{CaO}$ . 23  $\text{Na}_2\text{O}$ . 120  $\text{SiO}_2$



So, before we go into details it is important for you to know that how actually the molecular formula of the glass that is calculated and estimated and across the community in the globe, the molecular formula for a particular type of glass is almost universal. So, once we talk about the flat glass, window glass, the soda-lime-silica type

Then, if you go by the weight percent so, in 100 grams silicon dioxide will be 72, sodium oxide will be 14 percent, calcium oxide will be 9, magnesium oxide will be 4, and aluminium oxide will be 1. And I hope that you remember that, what is the function of what and what is the function of  $\text{SiO}_2$ ? We all know that this is a good glass former.

Now, what is the function of this alkaline oxide that you must know, that it basically reduces the melting temperature of  $\text{SiO}_2$ . Now, what is the function of aluminum oxide or magnesium oxide in the glass that you must remember and if you if not then you collect it that what is their function.

So, this soda in oxide form never it is used in glass formation. So, the term that is used is soda means sodium oxide and then lime that is calcium oxide and silica is silicon dioxide. So, in the flat glass all the manufacturer they use the same kind of formula. But actually they do not use the compound which is sodium oxide  $\text{Na}_2\text{O}$  or calcium oxide.

So, the composition you should roughly remember that, what is the composition of which type of glass? And then you can estimate what is the molecular formula of this

particular glass composition for example, you need to know what is the molecular weight of silicon dioxide and all other relevant material.

And then, this weight percent you divide it with the molecular weight and you get this numbers right 72 divided as 60.1 and you will get this numbers then, whichever is the lowest number then you divide everything by the lowest number.

So, you come up with for each corresponding oxide what are the values here and then you write a molecular formula aluminum oxide is 1 then magnesium oxide of 10 and silicon dioxide 120. So, this molecular formula is used across the glass community for flat glass composition.


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Raw materials required in soda – lime silica flat glass Batch calculation

Material	Alternative name	Theoretical formula	Oxides	Fraction	Batch	Purpose
Aluminum hyd.	Hydrated alumina	$Al_2O_3 \cdot 3H_2O$	$Al_2O_3$	0.654	1.531	
Limestone	Calcium carbonate	$CaCO_3$	CaO	0.560	1.786	Source of CaO
Lime, dolomitic	Burnt dolomite	$CaO \cdot MgO$	CaO MgO	0.582 0.418	1.720 2.390	Source of CaO and MgO
Soda ash	Sodium carbonate coml.	$Na_2CO_3$	$Na_2O$	0.585	1.709	Source of $Na_2O$
Sand	Glass sand, quartz	$SiO_2$	$SiO_2$	1.000	1.000	

$CaCO_3 (s) \rightarrow CaO (s) + CO_2 (g)$

- The fraction of CaO (mol weight 56.1 g/mol) in  $CaCO_3$  (mol wt 100.1 g/mol) is 0.56. For each kg of limestone added in the batch CaO will be 0.56Kg.
- Using the above raw material we can determine the batch composition of 1kg melt



Now, what are the raw materials that will be required? Aluminum hydroxide that is used that material and alternative name that is hydrated alumina the formula you need to know, and what is the main oxide component you need to know, what is the fraction of that oxide component inside that raw material, that you need to know.

So, same thing applies for the raw material of calcium oxide which is limestone, but calcium oxide also comes from dolomite because you need to use magnesium oxide. So, in magnesium oxide is there in dolomite and along with that calcium oxide is also there and burnt dolomite in fact is used. So, you need to use this raw material then, you need to

use this soda ash, which is sodium carbonate commercial variety so, that will give you sodium oxide and sand this is quartzite rock or glass sand silicon dioxide that is used.

So, now during batch calculation you will have to estimate what are the reaction involved in it. Calcium oxide, calcium carbonate if you are using then it will undergo calcium oxide plus carbon dioxide. If you are using dolomite the same thing is there because, this burnt dolomite of course is having calcium oxide and magnesium oxide so, directly one can use.


But for calcium carbonate you need to know what is the yield. So, here the yield is 56.1 gram per mole because carbon dioxide is going in the ambient back. So, calcium carbonate is having a molecular weight 100.1 and you are getting 0.56 of it for the calcium oxide form. So, for each kg of limestone it will have roughly about 0.56 kilogram. So, using the above material raw material which has been shown in the table, we can determine now the batch composition for 1 kilogram melt.

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**The batch calculation for Soda – lime – silicate glass** Batch calculation

- Instead of pure oxide natural raw materials (Col 1) are used instead of pure oxides.
- Fraction of oxide in the raw material is given in Col 2.
- All manufacturers of say flat glass **use same formula**, **Molecular formula** :  $Al_2O_3$  10 MgO. 16 CaO. 23  $Na_2O$ . 120  $SiO_2$  (i.e Molecular weight = 10038 g. Therefore in 1000 g melt, oxide required (Col 4)
- Knowing fraction of oxide in the raw material, required raw materials are estimated in Col 5. Note that source of CaO is both from dolomite and limestone.

Raw material	Oxides supplied	Fraction of oxide	Weight of oxide required (for 1kg)	Weight of raw material in batch (g)
Limestone	CaO	0.560	89.42	<b>60.625</b>
Silica sand	$SiO_2$	1.000	718.46	<b>718.46</b>
Soda ash	$Na_2O$	0.585	142.06	<b>242.83</b>
Alumina hydrate	$Al_2O_3$	0.654	10.17	<b>15.55</b>
Burnt dolomite	CaO	0.582	39.84 (MgO)	<b>95.31 (contain 55.47 CaO)</b>
	MgO	0.418		



So, as I have said that instead of pure oxide natural materials that is used to make it actually commercially economic. So, limestone is used silica sand is used that is given in column number 1. Now, fraction of the oxide in the raw material is given in the column number actually it is column number 2.

Because, this is the oxide that you are getting out of this raw material and this is the fraction oxide that you are getting for example, in silicon dioxide you are getting the full amount. So,  $\text{SiO}_2$  is only there, but in soda ash also carbon dioxide will not be there so, you get what is the fractional amount. So, that is given in column number 3 it is not column number 2.

And all manufacture as I said they use the same glass formula so, this is the molecular formula of the glass. So, therefore, you can have a molecular weight. So, from 1000 gram melt, whatever the oxide is required that you can calculate back and that is given in column number 4. So, for 1 kg of melt what is the amount of limestone etcetera that is required?

Now, here when you calculate it back you need to know that what fraction of calcium oxide component you will be getting out of this limestone. So, you can work it out yourself that how much limestone you actually will need, but a bit complication is there in this particular case because calcium oxide is also coming from dolomite. So, you will have to take into account, otherwise it will be non stoichiometry we will add more calcium oxide.

So, that you will have to check what kind of raw material you are using, whether it is a single source raw material or if it is a multi source raw material. If there is a multi source raw material depending on the your requirement out of this molecular formula what exactly will be needing that you will have to calculate back.

So, I have done it for you, for this molecular unit and you can see that these are the weight of raw material that is required because, in industry will have to tell that how much limestone I will have to mix in the batch.

Because people will not understand or they will not be in a position to calculate the oxide content etcetera, they will only understand the language that how much limestone I will have to put, how much silica sand I will have to put. So, that for 1 kilogram of batch this is the actual table. So, please work it out.

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Processing bulk glasses

Side view

Temperature distribution

1700°C  
1600°C  
1500°C

Processing bulk glasses

Glass tank furnace

Top view

- Batch is loaded into the **hopper** (1-2 **doghouse**)
- Batch begins to melt (3)
- Melt temperature peaks in region 4
- **Throat** (5) two parts : melting end and the working region. In working region  $T = 1300^{\circ}\text{C}$  and the glass viscosity increases.
- Glass travel through **forehearth** ( $1000 - 1100^{\circ}\text{C}$ )
- **Regenerative chambers** (9) are used to preheat the fuel gases and air to increase efficiency

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And this kind of calculation this is usually done in undergraduate level ceramic engineering course and this is industrial stoichiometry a typical subject name that is given for this kind of batch calculation. Now, once you calculate the batch actually this is a side view of a sorry elevated elevation of a glass tank furnace and this is the actual glass tank furnace for processing of the gas.

It is a huge furnace as you can understand it is having very different zones which control the viscosity you know the viscosity is very important starting from the melting zone up to the working zone so, the viscosity is neatly controlled. And this is the plan view looking at the top that how the glass tank furnace will look.

So, batch is loaded into the hopper which is 1 and 2 and sometimes it is called as dog house in terms of the glass terminology then, batch begins to melt at region number 3 then melt temperature peaks in region number 4. So, the temperature profile is also shown on top.

Then, throat is a region where it is basically divided the glass tank in two parts in this region, the melting end and the working region. So, in the working region the temperature is typically 1300 degree Celsius and the glass viscosity of course increases progressively from melting up to this region.

Then, glass travels to the forehearth these are the forehearth some of them are mechanized and some of them are manually controlled and this forehearth is having a temperature about 1000 to 1100 degree Celsius. And there are regenerative chamber here so, that regenerative chamber are used to preheat the fuel gas in the air to increase the efficiency.

Because, here you need to have more temperature as you can see particularly in this region and this region it is cooling down. So, initially you can preheat here and here you need to cool down so, this flue air you can use here. So, that is the purpose of this regenerative unit.

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**Bubble formation**

**Bubbles:** SO<sub>2</sub> and CO<sub>2</sub> from the dissociation of carbonates and sulfates. Glass – refractory reactions.

Bubbles are eliminated by **fining**.

- Temperature of the molten glass is increased by about 150°C to reduce viscosity. Drift velocity of the bubble towards surface is increased.
- Addition of **fining agent** like Na<sub>2</sub>SO<sub>4</sub>  
 $\text{Na}_2\text{SO}_4 (\text{s}) \rightarrow \text{Na}_2\text{O} (\text{l}) + \text{SO}_2 (\text{g}) + 1/2 \text{O}_2 (\text{g})$ .
- Large quantity of gas bubbles coalesce with existing bubbles. Volume increases, easily float to the surface.

**Processing bulk glasses**

Bubbles

Bubbles

The slide features a video inset of a man in a suit speaking, and two images: one showing a glass melt with a bubble and another showing a close-up of many small bubbles.

So, once the glass is coming out from the so called forehearth then it is something like this and the major problem comes that it contains bubble, and the bubble is coming from sulphur dioxide and carbon dioxide form the dissociation of carbonate which you have used you have used in the raw material and also the glass tank furnace refractory and the glass melt they are interacting and it forms bubble.

So, the bubble if it is there in the glass then it is useless. Because, first of all the transparency will be lost and strength will be less already you know that, you know about the mechanical property details whatever I have covered earlier. So, this bubble will have to be removed. So, the process of eliminating the bubble from the glass melt



that is known as fining so, for that purpose temperature of the molten glass is increased by about 150 degree Celsius to reduce the viscosity.

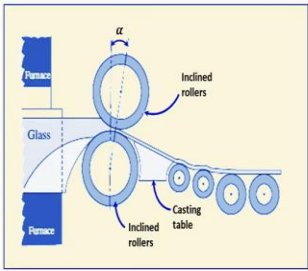
And once the viscosity is reduced then the drift velocity of the bubble increases. So, it comes towards the surface and then it annihilates or you can add fining agent like sodium sulfate. Now, so sodium sulfate they are dissociated with sodium oxide which is a glass component and sulphur dioxide is and oxygen they are generated.

So, you are generating more gas so, this large quantity of gas bubbles they coalesce and with the existing bubbles and the volume basically increases and they float to the surface and they are removed. So, this is a very important process the fining one to get rid of the bubble from the glass.

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**Flat glass fabrication** **Processing bulk glasses**

**Rolling**



- The molten glass flows from the tank furnace over a refractory lip and between a set of water – cooled rollers where it solidifies into a continuous ribbon.
- Ribbon is transported over rollers into a tunnel like annealing lehr furnace.
- Inside the lehr glass ribbon is heated 600 – 800 °C and then cooled gradually to reduce the internal stress of the glass.
- The glass thickness is controlled by a combination of (i) rotational speed of the roller, (ii) spacing between the roller and (iii) the glass pull rate (0.5 – 5 m/min). Glass thickness is 3 – 15 mm, and up to 3.6 m wide.

Continuous casting of flat glass by rolling

*(A small inset image of a man in a suit is visible in the bottom right corner of the slide.)*

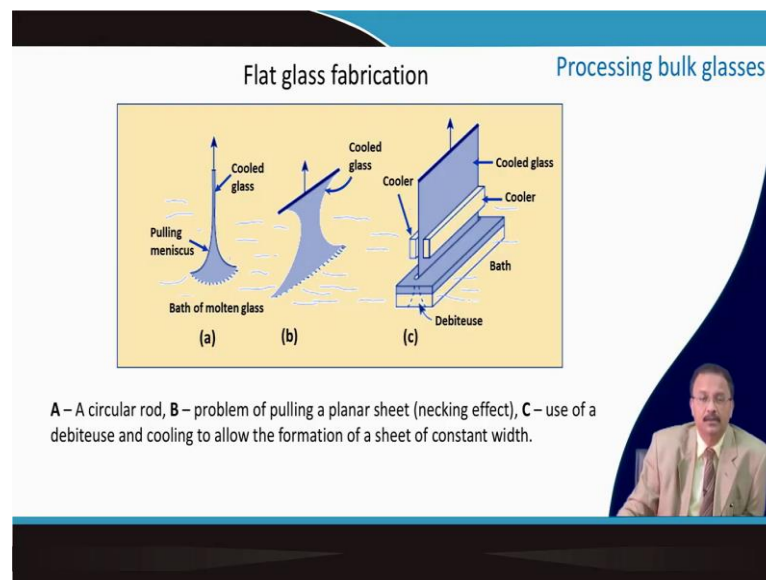
So, now the molten glass flows from the glass tank furnace over a refractory we call it is a refractory lip between a set of water cooled roller so, continuously the viscosity increases and here it solidifies into continuous ribbon and this ribbon is transported. So, actually this ribbon is transported through this Lehr so it is a continuous process.

So, glass is coming from forehearth and through this going viscosity is increased and this is forming a ruler and then it transferred to a Lehr furnace. And in Lehr furnace this is again heated at 600 to 800 to get rid of the strain involved already discussed when I was talking about the glass in I guess fifth fifteenth lecture well three lectures we have

included describing the glass. So, there it is mentioned about the Lehr furnace function of the Lehr furnace.

So, then it is cooled progressively so that the stress is relieved. The glass thickness is controlled by a combination of this rotational speed of the roller then, the spacing and also the pull rate typically 0.5 to 5 meter per minute pull rate is maintained and the glass thickness it can be 3 to 15 millimeter and usually it is about 3.6 meter wide.

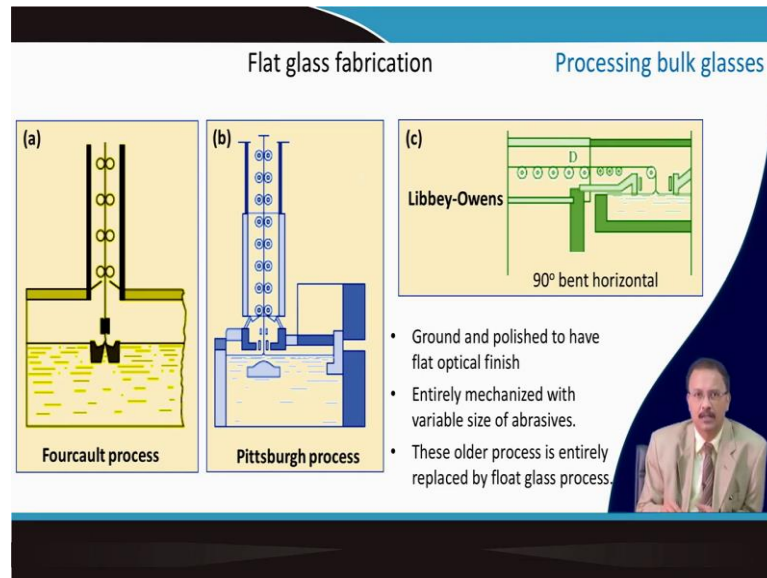
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Another flat glass fabrication is a drawing process. So, a circular rod you know that this is a viscous mass so with a circular rod you can make a tube, but for the planar glass you know that this problem of a necking is always there because, if you want to draw it while it cools it forms this kind of necking.

So, it is used a debiteuse that is a cool debiteuse is used to form a sheet here and then, it is a constant width glass can be prepared from the glass bath, but these are all old technology nowadays it is not followed.

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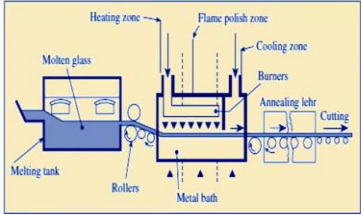


So, it has a different process name the Foucault process or Pittsburgh process depending on the place where exactly it was generated and these are all vertical process of drawing, but this Libbey-Owens process here the glass is 90 degree bent here right. So, this is 90 degree bent and then you form a flat glass bents. So, since either it is going through roller or most of the cases it is going through roller as you can see.

So, it should be ground and polished to have a flat optical finish, and entirely it is mechanized variable size of abrasive use progressively first the coarse one then the medium one then the final one abrasive finer particle size is required for good polishing. And these are all older process and entirely they are replaced by float glass process.

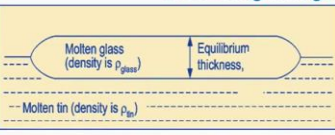
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### Float glass fabrication



- Tin oxidation is a problem, hence 10%H<sub>2</sub> – 90%N<sub>2</sub> ambient is maintained
- Reasonably flat surface of glass.
- Thinner glass – the pulling speed is increased
- For thicker glass spread is restricted

### Processing bulk glasses



Gravity and surface tension of molten glass leads to

Equilibrium thickness ( $h_e$ ) is given by

$$h_e = \left( \frac{(\gamma_{Gv} + \gamma_{Gt} + \gamma_{Tv})2\rho_t}{g\rho_G(\rho_t - \rho_G)} \right)^{1/2}$$

$\gamma_{Gt} \sim 1\text{J/m}^2$ ,  $\gamma_{Tv} \sim 0.68\text{J/m}^2$ ;  $\rho_t = 7.5\text{g/cm}^3$ ;  
 $\gamma_{Gv} \sim 0.35\text{J/m}^2$ ,  $\rho_G \sim 2.5\text{g/cm}^3$ ,  $h_e$  is estimated to be **9 mm**

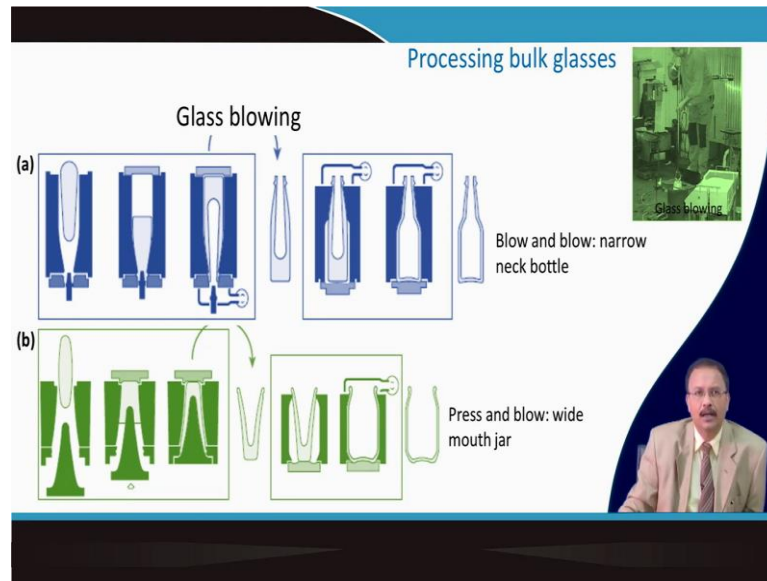
And in case of float glass the glass is coming from this region and then it is the lay the roller usually this is similar kind of thing, but then it is spread on molten glass sorry molten tin. So, molten tin it is floating here and once it you spread this one, you see that it has its own gravity gravitational force and also the surface tension right.

So, this two kind of force is operative it is something similar to this. So, molten tin is having some kind of density and glass is suddenly lighter than that. So, it will always float, but due to the action of these two different types of force it will have a equilibrium thickness. Equilibrium thickness can be calculated by this simple relation so; here different types of surface energies are involved.

So, this surface energy is between glasses and tin between your glass and air so, this you if you put this along with the density of the glass and the tin then the equilibrium thickness that you will get is roughly 9 millimeter. So, you can work it out you will get around 9 millimeter and actually people reports about 7 millimeter is the equilibrium thickness for this composition of the flat glass during float glass preparation.

So, thinner glass you can get by if you increase the pulling speed and for thicker glass if you want then the spread will have to be limited. So, above 25 millimeter thick glass pan you can generate. So, tin oxidation is a problem here so, this ambient should be maintained which is 10 percent hydrogen and 90 percent nitrogen ambient and here one advantage is that very good flatness you can get out of this float glass process.

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So, glass blowing is another so that is all about the flat glass process, otherwise you can use glass blowing and here in case of glass blowing you used gob this is called glass gob. And first due to gravity it falls here and then you apply a air pressure and the mould is something like this. And then you get a glass article out of the mould and again you blow inside a mould. So, this is blow and blow process to get a narrow neck bottle like this.

So, glass blowing is very important concept earlier people used to do it manually and still some people are there who can do manual glass blowing and that is particularly useful for having different types of joint in chemistry lab to make a nice system for chemical process. So, that is one thing that is there; and otherwise it is all mechanized nowadays.


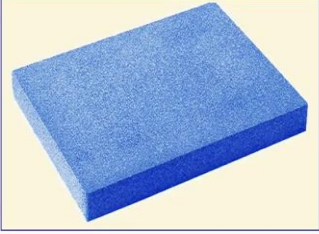
And the second one is press and blow process; this is actually useful for wide mouth jar. So, here initially you are pressing it and get this article in the form pre form in this way this particular shape and then you put it in the mould and blow it so it is fluid enough so, that it covers this space and you get a wide mouth bottle. So, this is a by blowing process.

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**Foam glass**

The opposite approach of fining is adopted to make foam glass. Additional gases or gas – bearing materials are added to the melt producing a large quantity of bubbles.

One major application of foam glass is in the manufacture of insulating panels for buildings. Insulating panels made from foam glass can be light, rigid and good thermal insulators. As usual for porous materials, the thermal property is due to the low heat conductivity of the bubbles (i.e pores)



Foam glass is important where it is opposite to the bubbling effect because, as I told that bubbling is not good, but for some purpose we need bubble in the glass. So, the transparency is lost because lot of air bubble is there inside the glass and due to that lot of light scattering will take place so, it is no longer a transparent material, but it is a highly porous material and it is light weight.

So, this is an opposite approach for fining is adopted to make a foam glass and additional gases or gas bearing material they are added to the melt to produce large quantity of bubbles.

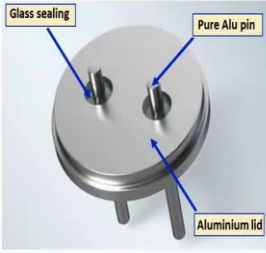
So, one major application of this kind of foam glass is in manufacturing of insulating panel for the buildings. Insulating panels made from the foam glass can be very light, rigid and good thermal insulator. As usual for porous materials, the thermal properties is due to the low heat conductivity of the entrapped air that is there inside this foam glass.

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### Sealing glass

Special glasses have been developed that are used as the “glue” usually between another glass and a metal. The main challenge is engineering  $\alpha$  while controlling chemistry and keeping the sealing temperature reasonable.

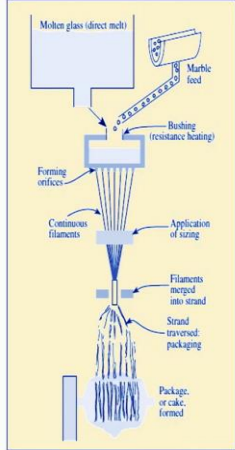
- Borosilicate glass is used to seal glass to W
- Borosilicate glass is used to seal glass to Mo
- Pb glass is used if electrical insulation of a joint is critical.



Another one is a sealing glass this is very important because in certain electronic application you need to insulate this two conductor so; there is a glass seal here in between. So, this inner material is glass seal and this is aluminium pin and this is also metal body. So, special glass have been developed for this kind of sealing that used basically as a “glue” between another glass or glass metal.

The main challenge is to control the thermal expansion mismatch between this two material so, that it does not crack. So, usually borosilicate type of glass that is used and particularly to use with tungsten it is possible, it is used to seal molybdenum lead gas is actually used for electrical insulation like this. This is a very critical application and not all the compositions are very widely reported because, that is a secret because these materials are quite interesting to have.

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The diagram illustrates the fabrication of glass fiber. It starts with molten glass (direct melt) being fed through a Marbic feed into a bushing (resistance heating). The glass then passes through forming orifices to create common filaments. These filaments then undergo application of sizing. The filaments are merged into a strand, which is then traversed and packaged. Finally, the package or cable is formed.

### Fabrication of glass fiber

	E glass	C glass	S glass
SiO <sub>2</sub>	55.2	65.0	65.0
Al <sub>2</sub> O <sub>3</sub>	8.0	4.0	25.0
CaO	18.7	14.0	—
MgO	4.6	3.0	10.0
Na <sub>2</sub> O	0.3	8.5	0.3
K <sub>2</sub> O	0.2	—	—
B <sub>2</sub> O <sub>3</sub>	7.3	5.0	—

E is for electrical. It is a good insulator  
S is for higher stiffness and strength  
C glass has high CaO contents and yield corrosion resistance in acid and alkaline environment



Finally you know about the glass fibers, which we have used for the reinforcement and this is in short that how the glass fibers are made. There are various compositions of these fibers and we call its E glass that is for the electronic purpose where it is having a good insulator. So, you take the glass melt or you can use the glass marble here in the molten form; and then there is a turret here.

So, here the glass fibers are drawn and after you draw the fiber then you apply a sizing. So, that sizing is a polymer kind of coating you know that this is important because, glass fiber as such their surface should be protected. If surface is having a small crack, that will destroy the strength characteristics of this fibers.

So, sizing is done with the polymer coating and then, they are wind up winding in a spool for the reinforcement purpose. So, this is a simple technology that is adopted for glass fiber fabrication. So, this E glass is for insulators and S glass is for higher stiffness and strength.

So, this S glass you can see the composition wise they are different here and this is having higher stiffness and strength and that the other one is actually we call it is a C glass, where calcium content is more. So, as you can see calcium content is relatively higher in C glass here



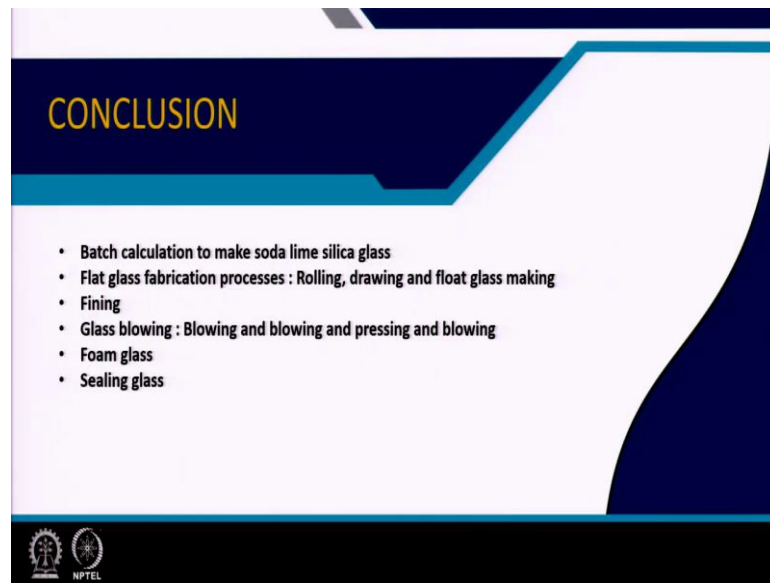
And in case of S glass, actually this is magnesium oxide is more this magnesium oxide is more as compared to this one and this C glass they are corrosion resistant particularly resistant to acid and alkaline environment. So, depending on the composition different types of I mean depending on the use different types of composition of glass fiber that is used.

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So, the reference for this particular chapter is a book by Barry Carter and M. G Norton Ceramic Materials and Science and Engineering, Chapter number 26, where it talks about the processing of the glass and apart from that the book by Barsoum Phillips and Pampuch there those are also a good reading material for this part of the lecture.

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So, in this particular lecture we talked about the batch calculation to make soda lime silica glass, flat glass fabrication process including rolling and drawing which are obsolete nowadays almost it has taken over by the float glass making process. The concept of fining sometimes it is required sometimes it is not required.

You intentionally do not do fining opposite to the fining you do for making the foam glass then, we introduce various techniques for glass blowing including the blowing and blowing or blowing and pressing and blowing kind of concept and finally, sealing glass is introduced along with the glass fiber and different compositions.

Thank you for your attention.