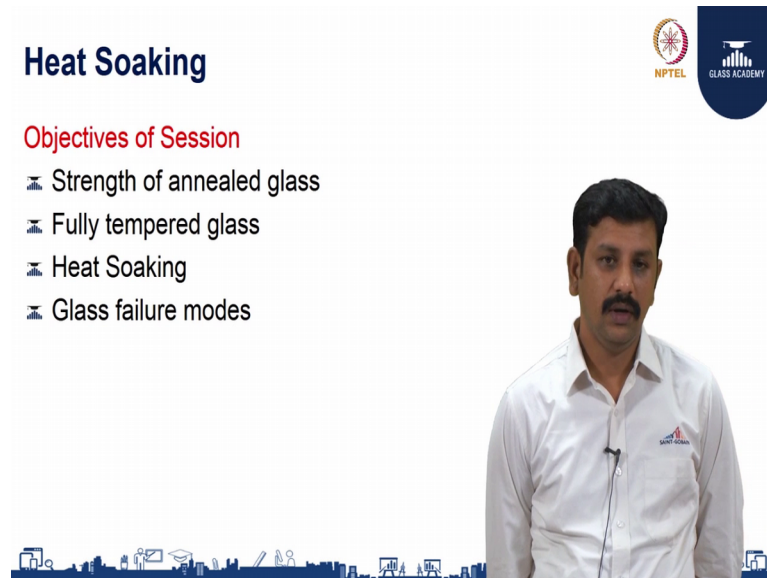


**Glass Processing Technology**  
**Prof. Mr. Muthukaruppan**  
**Department of Materials and Metallurgical Engineering**  
**Indian Institute of Technology Madras**

**Lecture – 57**  
**Heat Soaking\_ Part I**

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**Heat Soaking**

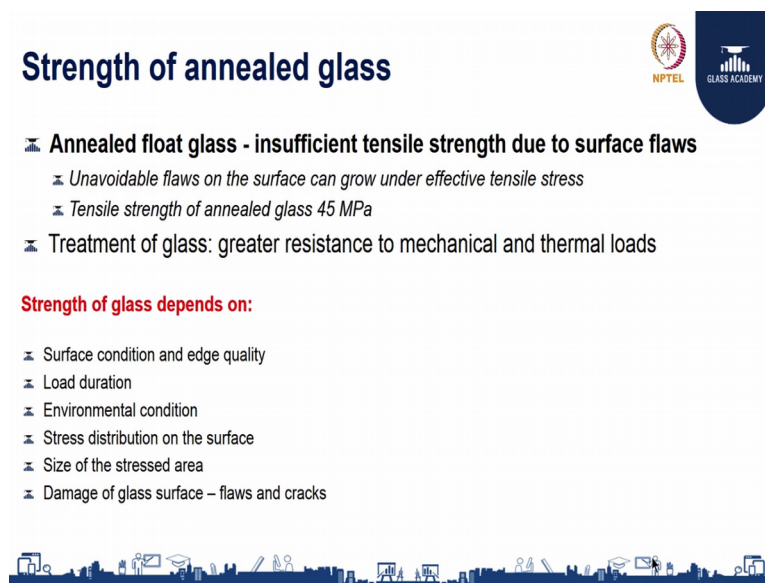
**Objectives of Session**

- Strength of annealed glass
- Fully tempered glass
- Heat Soaking
- Glass failure modes

The slide features a video of Prof. Mr. Muthukaruppan, a man with a mustache wearing a white shirt, standing in front of a white background. The slide also includes the NPTEL logo and the Glass Academy logo in the top right corner, and a decorative blue silhouette of a city skyline at the bottom.

Good morning we will go to Heat Soaking. This is a session about heat soaking of glass. So, objective of the session will be to understand strength of annealed glasses, you know annealed glass you are going through the float process, the annealed glass is producing a float bath and it is cooled it has very slow rate in annealing layer of float. The purpose of annealing of the glass is to have uniformity across core and center of the glass and we will also learn about what is fully toughened tempered, tempered glass and how do we go through this tempering process. And what it makes an impact with it then we will go in detail on the heat soaking and we also learn about the glass failure modes.

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**Strength of annealed glass**

- **Annealed float glass - insufficient tensile strength due to surface flaws**
  - Unavoidable flaws on the surface can grow under effective tensile stress
  - Tensile strength of annealed glass 45 MPa
- Treatment of glass: greater resistance to mechanical and thermal loads

**Strength of glass depends on:**

- Surface condition and edge quality
- Load duration
- Environmental condition
- Stress distribution on the surface
- Size of the stressed area
- Damage of glass surface – flaws and cracks

So, to start with we will start with the strength of annealed glass, the strength of annealed glass is basically it has an insufficient strength tensile strength at the middle where because of the surface glass. We know that the annealed glass will go through a tin bath. So, it is floating process of float making is having the glass floated in the tin bath. We take out from the tin bath and put it on the left out production and then further processing are done in annealing manner. The bottom side that is the tin side of the glass we have a micro flask; the tin size of a glass is always having a micro flask compared to the air set of the glass.

So, the there are 2 stresses which we normally talk about one is the tensile stress on the glass which is 45 mega Pascal and also there is a stress which is on the surface we measured it using glass instrument and it is 2 to the power 25 mega Pascal, the treatment of the glass will make it more greater resistance to the mechanical and the thermal stresses. So, basically the glass strength depends on the following parameters, one is the surface condition and the edge quality.

So, the surface condition when you talk about surface condition it is a about the surface this, suppose the there is an acid edge glass or a sand (Refer Time: 02:13) of glass. So, the stress levels are quietly low and also on edge quality where we cut the glass that there is a possibility of stress getting relieved. So, where the cut quality is determines the edge quality the edge quality can be maintained by having proper grinding of glass.

The grinding can be from the venture of arising simple arising to (Refer Time: 02:29) edges the load or to have a phrase grinding, the thicker the glass is it is better to have full phase grinder. Second is on load duration when you install in a building it also depends on the load duration how much time the load is on the glass and also environmental condition.

When we talk about the environmental the extreme conditions which work it makes the different, basically the environmental conditions with the high humidity with acidic and alkaline environmental conditions and on sometimes with the high wind velocity, normally the wind flow and the wind load is what it depends; stress distribution on the surface of the glass also determines it is differences and also the size of stressed area. So, if your glass is relatively smaller or a bigger one it has a surface stressed area, this size of the stressed area also determine the strength of the glass, then particular on the glass surface if it has flaws, cracks, chips all that will determine the damage the things.

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**Why temper (strengthen) glass?**

- ⊖ Tempered or toughened glass is a type of [safety glass](#) processed to increase Stress to 4times of annealed glass
- ⊖ Principally similar to "prestressing" - methods in structural engineering;
- ⊖ Improve breakage performance due to small, blunt pieces/splinters – so called safety glass (a tempered glass);



**Typical Tempered Glass Break Pattern**

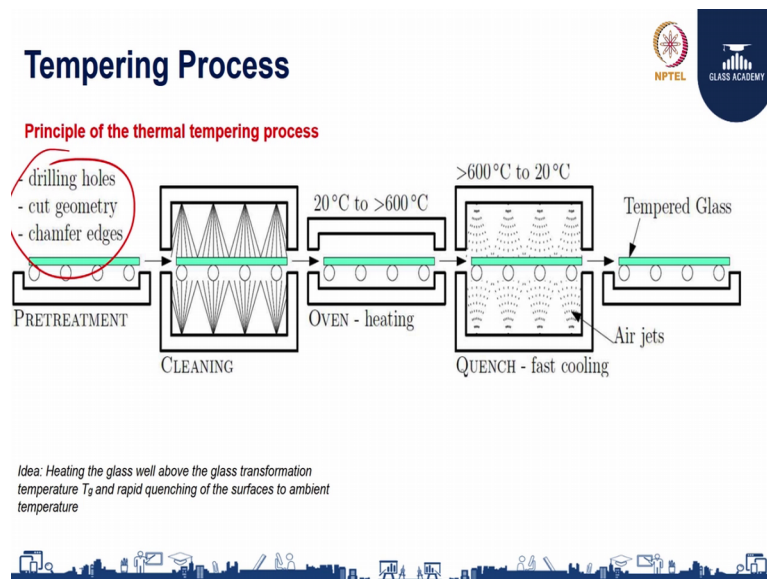
"Because of the break pattern, tempered glass is much more likely to evacuate the glazing system immediately upon breakage."

The slide includes logos for NPTEL and Glass Academy at the top right, and a decorative city skyline at the bottom.

Now, why do you temper a glass basically we temper a glass to improve it is ability to withstand the stress. So, normally the toughened glass is a fully tempered out of a toughened glass, so it has 4 to 4 times the stress of a annealed glass. So, roughly it will take around hundred and 10 to 120 mega Pascal forces principally it is similar to the any pre stressing process which we use in steel iron cast iron and all that and it helps us to improve the breakage pattern, in case if it is an annealed glass you can see in the picture.

The breakages will be sharper in nature and then it will have an injury to a human and so it is not normally a safe glass. So, the tempered glass will have a breakage which is very small in nature and it is very blunt. So, normally it is measured by having a 5 centimeter by 5 centimeter square and if you count the number of breakages it will we have to get 40 pieces. So, this is how the stress glass will qualify as a fully toughened glass, this is called safety glass basically, because of the minute particles and the blunt nature which will be relatively low harmful to the humans.

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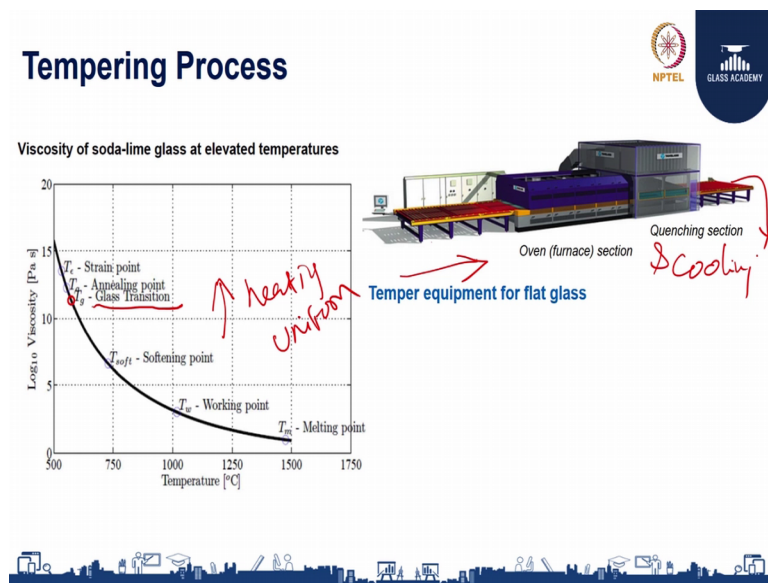
So, now, we need to understand how it is done. So, principle of a tempering process there are 4 stages in the tempering basically first the pre cuts anything we need to do that is drilling of glass geometry, cutting of the glass, chamfering the edge of the glass the precise size, after tempering you will not able to do any cutting of glass. So, the pretreatment everything which is selected to that including drilling holes cutting glasses to the required sizes treating the edges everything has to be done before that. And (Refer Time: 05:09) cleaning of the glasses is very critical because, chips anything will get fluxed in to the glass and forms the permanent defect on the glasses.

So, glass has to be cleaned as much as possible this applies both to the normal glass and the circular glasses and the UV coated glasses. Then emissivity in the glass makes the glass tougher to temper, lower the emissivity of the glass it is more tougher to temper

because, of the reasons that this emissivity lower emissivity means it has higher reflections towards the in infrared radiations.

Now next stage it goes to an oven where it is getting hitched up, the normal temperatures will be in the room temperature with 30 degrees and it has to cross the TG that is a glass transient temperature which will be greater than 650 600 degree Celsius. From 600 degree Celsius it is cooled in cooled very fast that is called quenching.

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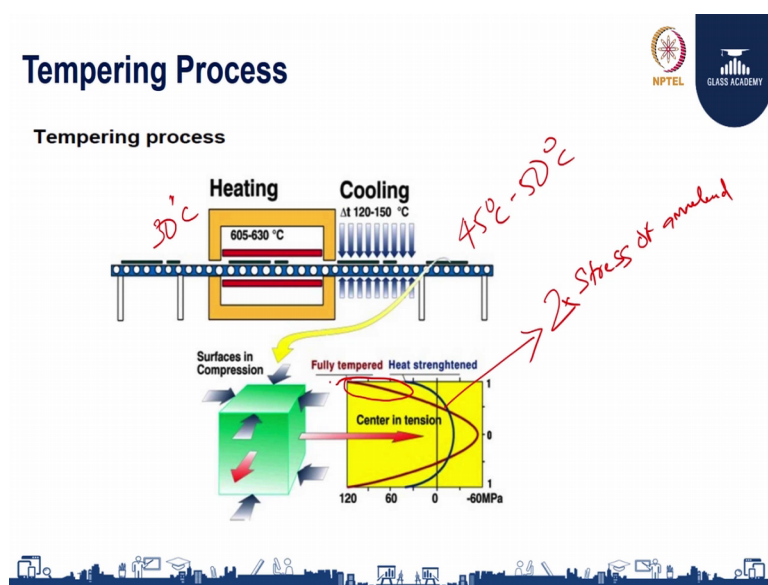
So, this is basically done to have create a difference between the top and the core temperature, when we quench the glass faster the top cools faster and the center remains hotter. So, this implies a compressive stress on the top of the glass which makes the glass tempered and what we have seen in the last lecture, it makes the glass to break into smaller pieces when it is subjected to that.

The clear thing idea here is to the heating of glass has to be well above the glass transformation temperature and it should be rapidly quenched because, the heating of glass has to be above the glass transformation temperature and it has to be rapidly cooled to ambient temperature. When you say rapidly cooled the normal quench time is only 7 percent of the heating time, so it is roughly for thickness of 6 mm glass the heating time will be anywhere in the range of 250 to 300 seconds and the quenching of time it takes place only 20 to 25 seconds. After that it is only cooling of glass to the room temperature which is only for handling and other related issues.

So, temperature which I was talking to you about the glass transition temperature, this is the key temperature any glass heating has to be crossed the heating of glass has to be cross this temperature and the heating should be as uniform as possible, heating should be uniform to make sure that glass is flat after heating it. So, key agent has for soda lime glass which we will talk about we generally call total glass that heat in glass transient temperature will be close to 580 degree Celsius and we normally measure the air temperature in the tempering in the sense. So, normal air temperatures will be around 650 degree Celsius, so that it ensures the glass temperature as known after the glass of coming out of the heating zone will be measured using a pyrometer to understand a heat distribution.

The quality of tempering depends on the uniformity of the heating and how uniform you are able to cool in the heat it in the tempering process it is more critical for it is quality of both the optical and the tempering quality. So, this is the typical tempering machine normally glass will be loaded in this direction and once the glass is loaded it goes to the oven where we have a electrical heaters. Now this will heat the glass from room temperature to 650 degree Celsius and it exists the temperature the quench zone and cooling zone, it is also quenching and cooling. So, the first 20 seconds of it will be the vital period where we have a quenching process and then it is cooled to the room temperature and gets unloaded from this (Refer Time: 08:29).

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So, if you have seen how it performs to the glass, the glass loaded will be in the room temperature 130 degree Celsius and then when it goes to the heating temperature the glass temperature will be roughly 605 to 630 degree centigrade at the surface of which we measure. And then it is cooled first to, cooled at faster rate in quenching area and then finally cooled to the room temperature in roughly 45 degree Celsius to 50 degree Celsius where it can insert and then handled manually.

When we cool that as we already discussed there is couple of things which will happen, the top surfaces of the glasses will get cooled up faster and the center term glass will remain hot still. So, it means the top will be lower in size compared to the center which in later stage you may gets cooled, it forms compressive over the surface and the surfaces remain compressed in there. So, there are 3 graphs in when you see there are 2 majorly which is being defined one is heat strengthened glass, the heat strengthened glass and has a same principle of heating where heat strengthened glass is similar to the toughened glass, but it is 2 times the stress levels of annealed.

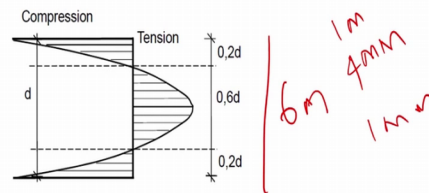
Twice x the stress of annealed glass, this is the intermediate glass which is available in market this is not called safety glass, but it is better to have case strengthened glass also in case of façade applications. So, when we compare the stress levels of both you can see a difference in the curve, so fully toughened glass will have a more compressive strength at the edges which makes the glass breakage into particles of sizes less than 1 mm or equal to 1 mm of glass.

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## A. Fully tempered glass



- Quenching (fast cooling) with air blown over both sides of pane
- Cooling and stiffening first on the surface, delayed cooling and consolidation of the core @ internal stress (parabolic distribution)
- **Surface in compression (90 – 150 MPa), core in tension**

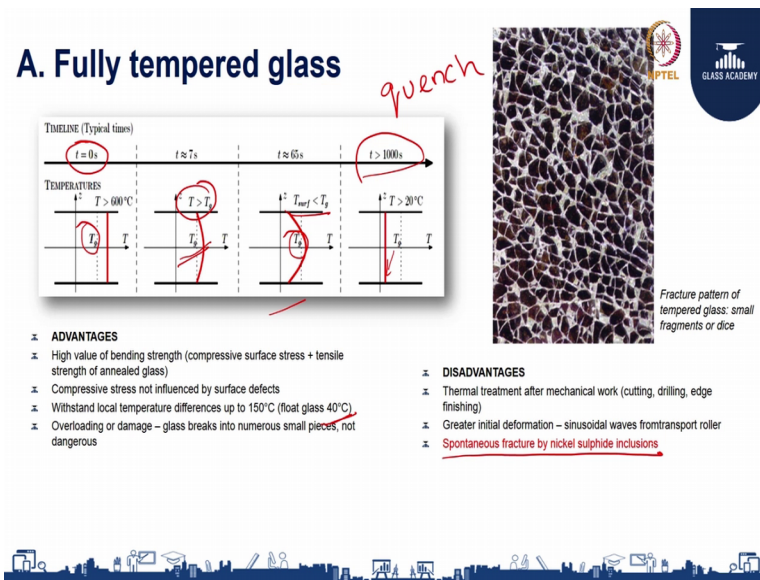


So, we recap what we have discussed in the fully tempered glass of quenching is makes a difference. So, heating process or fully tempered glass and the heat strengthened glass remains same. So, we heat the glass above the glass transient temperature and quench normally if it is the air blow, now if you have hot air, fast air blow from the both sides of the band from the top and the bottom and the distance that which you blow the air depends on the thickness of the glass and the stress load if you are able to measure in it, then the cooling and stiffening of the top surface will happen.

Now, the delay cooling in the center creates an internal stress which is you have seen in the parabolic distribution, we can again see in the parabolic distribution if you take a thickness one axis if it is a thick mm glass roughly 4 mm we will have the certain tension and last 1 mm at the top and 1 mm at the bottom we will have the compressive stress. So, this is the distribution which is roughly estimated for the fully toughened glass.



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So, when you take a time line, when time line versus the progress of the glass or the stress level change in the glass. So, the first graph shows you the time 0 that is at the start of furnace and the temperature is less than the TG temperature, the TG temperature is measured glass transition temperature. So, after 7 seconds when it comes out of the furnaces, this is only the quench graph, the graph is meant for only quench not for the heating.

So, once it is away from, come out of the furnace which will be normally greater than 600 degree Celsius and TG is greater than the 600 degree Celsius it comes to the next stage. Where after 7 seconds the top surface you can see is less than the TG value, so on the bottom surface both the surfaces are less than the TG value and center still remains in the TG and after 60 phase seconds.

You can see that top surfaces and the bottom surfaces is much below the TG and there is no further stresses possibility and the center core still remains above the TG values. And when you take end of the cycle may be after sometimes after you get stabilized cycle after complete cooling and unloading of the glass, the glass temperature will be much below then the TG and the surfaces will be remained constant, it means all compressive forces which is there is given to the glass.

So, there are couple of advantage of the tempered glass, so one is this high value of the bending strength when we put the glass in the bending machines and the above the glass

to bend that has a combination of surface compressive force and tensile force, which makes it highly safe glass. Second the compressive stress is not influenced by the surface stress, even if you have a surface stresses the compressive stress will not allow the curved surface stress the surface cracks to go inside a flask to go inside and make it difficult.

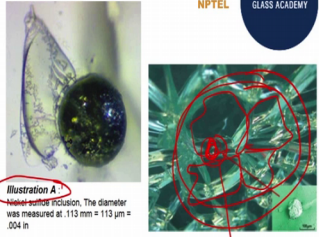
So, the breakage part is fully avoided unless we will exceed the force of grid at 100 mega Pascal than only glass, can natural fracture is less in temp surface. It can withstand local temperature up to 150 degree Celsius change in temperature and any float glass will be withstand normally 40 degree Celsius. When you compare to that the toughened glass can withstand up to a change of 150 degree Celsius in the atmosphere and any overloading as or damaged in the glass will be broken into small pieces which is completely have safer further people were in and around them. So, this is the fracture pattern we have been talking about all these times and there are couple of disadvantages also with this one is this thermal treatment after the mechanical work.

So, we cannot do anything after the glass is tempered, once the glass is tempered we cannot do anything on the glass and it is pre treated glass. So, it cannot be cut into the next size or anything. So, that is one disadvantage we have and normally it passes through a roller where it have a much higher deformation when compared to heat strengthen or annealed glass, optical distortion will be higher than annealed glass and heat strengthened glass. And most critically we have a problem of factor spontaneous breakages because, of inclusion of nickel sulfide in to the glass.

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## Spontaneous Breakages

- Spontaneous Breakages can Occur for Several reasons like
  - Surface damages, caused either before or after installation
  - Pitting, Welding
  - Foreign bodies such as nickel sulphide in the glass
- Nickel sulfide inclusion in the glass
  - A tiny metallic ball in the glass as you see in Illustration A,
  - Illustration B shows typical particles and the break pattern that results if the glass fails because of the particle.. - 'Butterfly pattern'
  - These Butterfly or ear lobes' are 2 - 6 sided particles
  - This spontaneous glass breakage happens all by itself, however Small external force provides just enough additional force to tip the scale and break the glass.



**Illustration A:** Nickel-sulfide inclusion. The diameter was measured at: 113 mm = 113  $\mu$ m = .004 in

**Illustration B:** "butterfly" failure mode

How big is the nickel sulfide inclusion?

	Small NIS Inclusion (dia)	Large NIS Inclusion (dia)
Millimeters (mm)	.05 mm	.1 mm
Micrometers ( $\mu$ m)	50 $\mu$ m	100 $\mu$ m
Inches (in)	.002 in	.004 in

*analysis*

Now, we will go in detail and understanding the spontaneous breakages and nickel sulfide which is playing a major role in this and the way to come out of it. So, spontaneous breakages even though it is normally associable with nickel sulfide inclusion in the glass, there are several other reasons also. We can define is 3 ways, one is this surface damages or any other things surface cracks damages many things present in this or some pitching welding activities which happen in this area which had dimension surface at the point of usage or the foreign bodies such as nickel sulfide in the glass.

So, when you talk about nickel sulfide, so first we will go through this illustration A. So, as an example of nickel sulfide, this is a bubble which is this is a metal ball which is nickel sulfide in it and the size of the nickel sulfide is normally 110 to 110 micro meters. So, it is roughly point 1 or 0.11 micrometers which can cross at breakage. So, nickel sulfide is a tiny metal ball you can see from the figure and then in terms of breakages we have to confirm the breakages because of the nickel sulfide, you can see always a nickel sulfide breakages will be associated with the butterfly pattern.

So, we call it as a butterfly pattern or air lobe pattern, so when you closely observe there are 4 similar to the butterfly it will break and this is a portion of nickel sulfide which is inside it. This pattern represents there is a metal inclusions and metal can mostly it will

be nickel sulfide inclusions, the nickel sulfide gets into the glass basically from the raw materials we use sand as the basic raw materials for producing glass.

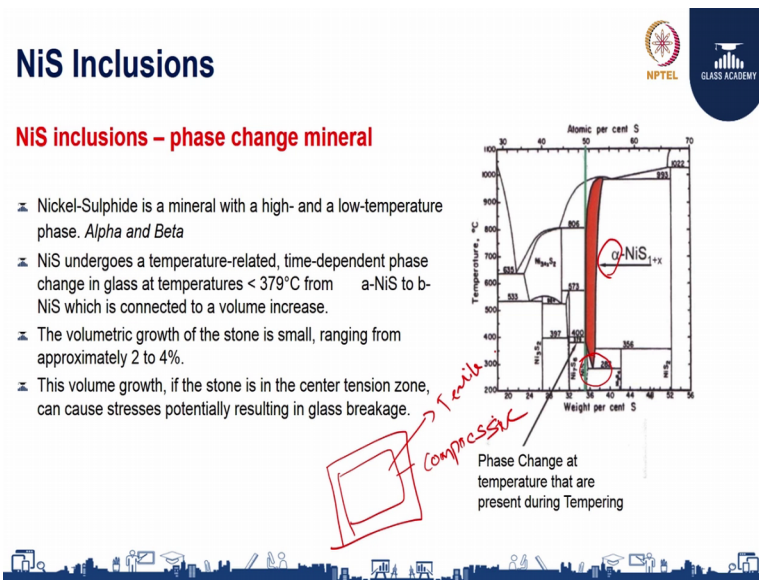
So, sand can contain metals heavy metals float by enlarge now has developed a more and more sophisticated technologies to remove as much as possible the metal elements into that. So, example they have a metal detector they have a magnetic detector they have more and more and equipments they use is nickel free equipments they use. So, more and more floats are modern floats are with less nickel sulfates corrupted.

But still there are possibilities of having nickel sulfide in to the glass which is of very very 0.11 0.1 mm size of nickel sulfide is. So, the pattern at which it breaks is normally the air lobe or butterfly pattern and this can be 2 to 6 sides, currently when the picture represent 4 sides it can be inclusion set butterflies it can be 4 sided it can be 6 sided in some cases and most of the cases this spontaneous breakage happens just like that without any interruption without people touching anything (Refer Time: 17:03) spontaneous breakages can happen.

But in some occasions this also happens with minute interruptions like opening a door, closing a door at higher force is also can earn to the butterfly pattern. But if it is broken because of nickel sulfide to confirm that we need to replace any breakages with this and then you have to remove this metal and analyze in the lab to confirm it is there nickel sulfide is present in it or not.

So, how big a nickel sulfide can be used usually it is smallest nickel sulfide inclusion can be of 0.5 mm and largest can be of 0.1 mm, we measure it in terms of micro meters, so 50 micro meters to 100 micro meter is a nickel sulfide.

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Why it breaks after tempering why it does not break in (Refer Time: 17:51)? So to understand that we need to go in little bit in inside the (Refer Time: 17:54). The nickel sulfide we have 2 phases one is the alpha nickel sulfide and the beta nickel sulfide. So, this phase changes happens at the temperature much lower than, so nickel sulfide has 2 phases one is the alpha phase and the beta phase and one phase that is a beta phase is not a stable phase. It is alpha phase this is stable phases, so because of this instability the change in phase will grow in volumes.

So for example, if you see a normal temperatures at which we can go it is 200 to 260 degree Celsius there is a much less than the 369 degree Celsius we can see here much higher graph. So, smallest volume can grow in high volumes, this growth in volume which means roughly 2 to 4 percent in the growth in volume will have a differential cooling curve than the glass.

If you cool the glass and we cool the nickel sulfide inside the glass, both will have a difference in cooling curve which results in the breakages of the glass. This volumetric growth even it is told to 2 to 4 percent of it, if you have to put into the glass sizes it causes the breakage particularly when the glass if you take a glass if it is the glass is having few centered normally we have the tensile area all the corners will be compressive and center will have the tensile area. So, any nickel sulfide in the tensile

area will tend to break in higher probability than compared to the in the compressive breakage.

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### **Summary:**

By the end of this video, you have learnt about:

- Strength of annealed glass
- Need for tempering of glass
- Tempering process
  - Fully tempered glass
  - Spontaneous breakage
  - NiS inclusions

