




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

Lecture – 58
Heat Soaking_ Part II

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Heat Soaking

- z Reheating Glass to 290°C +/- 10°C
- z Accelerate Phase Change of NiS
- z Glasses that survive the Heat Soak test either have no Nickel Sulfide particle present, or the particle is so small as to not cause a problem, or the particle resides in the compressive layer and again it should not present a problem in the future.
- z However, research by A Kasper, indicates that less than 1 break in 10,000 panes of glass is expected to occur after a Heat Soak.
- z Reference BS EN 14179-1 Standard



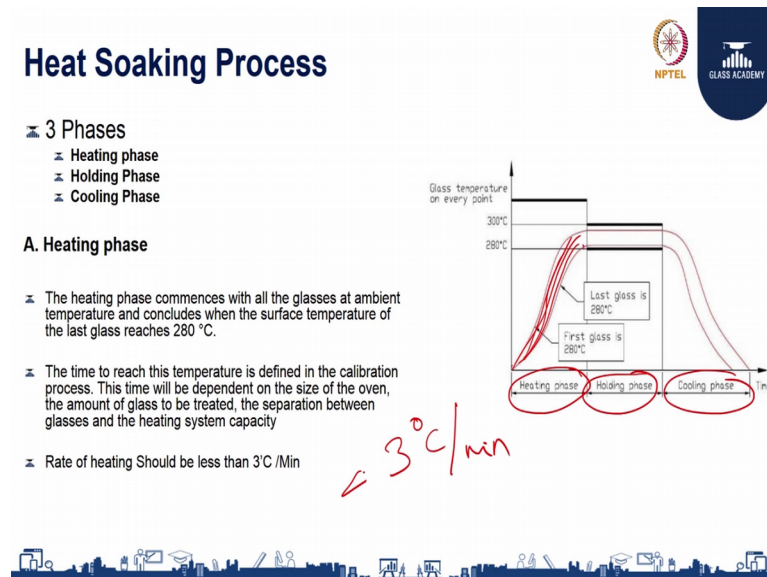
So, what to do in case of elimination of this, so that is where we are coming to our topic heat soaking. So, heat soaking is process it is a destructive process by which you reheat the glass in a oven. Here, you can see we have put the glass inside and reheat the glass into the oven at 292 plus or minus 10 degree Celsius. We accelerate the change of phase of nickel sulfide.

We want to a particularly the glass to change from one phase of nickel sulfide to make say (Refer Time: 00:50) nickel sulfide to it. If at all the glass has a nickel sulfide which is smaller in (Refer Time: 00:55), we ask this to grow in sizes, and it breaks. So, it is a destructive phase, destructive process by which in case the glass has a nickel sulfide. It breaks inside in the oven and the glass which comes out of it will have 0 or very very minimal nickel sulfide including of the glass or it will not break. It is in the phase where it is in the compressive area, it will not break in the future.

However, there are some researches there are n number of journals published on this heat soaking and the nickel sulfide. One gentleman called Mr. Kasper has done numerous

experiments with this. So, he still says even after heat soaking, there are probability of 1 breakages in 10,000 glasses 1 breakages for 10,000 glasses these are still probability So, we have a reference standard for it E N 14179.

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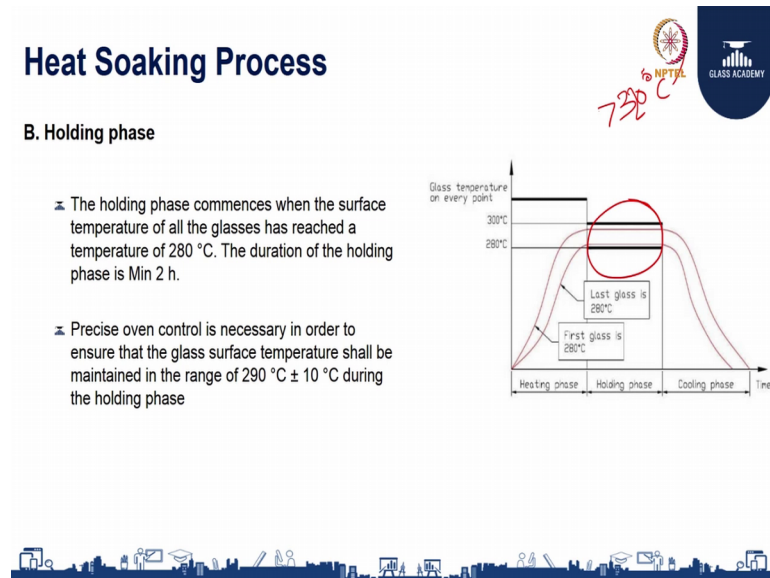


So, how does it (Refer Time: 01:49). There are three phases in this. One is the heating phase, second is a holding phase, and the cooling phase. So, in the graph, you can see these three phases indicating fully heating cooling. And in heating phase our idea is to heat the glass. We need to this tempered and cooled and kept it ready. We will keep the glass again from room temperature means we reheat the glass from room temperature to 280 degree Celsius. How do we measure the 280 degree Celsius? It is normally a batch operation, in a batch you put 10 thermocouples at different locations. To ensure all part of glass have reached it.

So, you can see 2 graphs. One is the; if you have a batch of 20 glasses, the first two glass all the thermocouples will be aligned. And the range all thermocouple has to the last thermocouple also has to reach more than 280 degree Celsius, means the temperature measured in the last glass also has to cross 280 degree Celsius until then the heating phase will continue. So, heating phase will continue until the last glass also reaches to 280 degree Celsius. Once it has cross 280 degree Celsius, then it goes to the next stage. The key challenge here is to have a oven calibrated to have a specific rate of heating, in a specific rate of heating is 3 degrees per minute.

So, it has to be heated at 3 degrees per minute max. So, it should be always less than 3 degree Celsius per minute heating. So, the precise control this is on the heating calibration of machines heaters and availability of the spaces, and other things. We will go in detail how it can be maintained, and how a calibration process works apart. Now, rate of heating should be less than 3 degrees per minute.

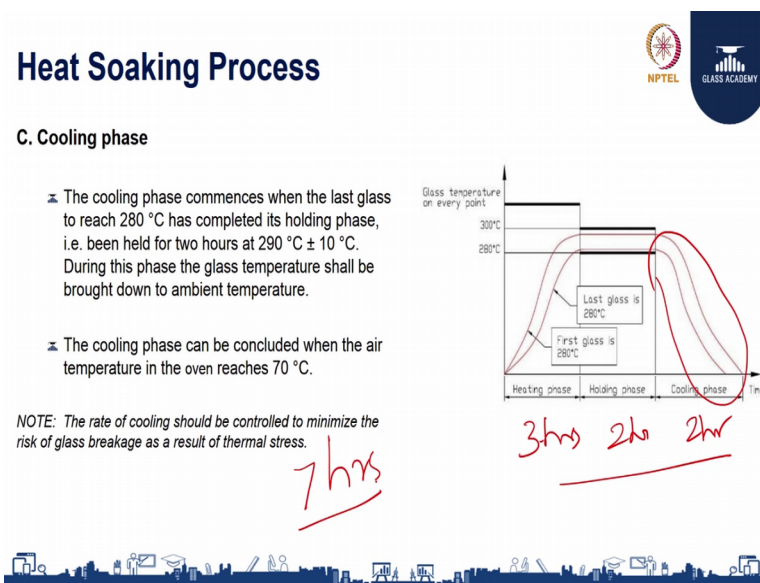
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Now, the second phase is what we are going to discuss here, this is on the holding phase. Here, we have to maintain the temperature at 280 degree Celsius constantly all the thermocouples means all part of glasses should be more than 280 degree Celsius always there will be small additional heat incremental heat even to maintain the temperature. And minimum should be maintained for 2 hours. There are several theories which has been note down (Refer Time: 04:02) several experiments to see how much is the hours of holding we have to have.

There are article saying we can have a faster cooling, faster holding rate means less than 2 hours there are sometimes it is more than 2 hours, but minimum 2 hours is what we recommend, normally the 2 hours becomes the standard now ok. The precise oven control is much more critical in this area, because if you do not control the temperature precisely, there are possibility of glass temperatures shooting more than 3 degree Celsius which is not acceptable. Any parking time greater than 320 degree Celsius is not acceptable. So, always it has to be less than 320 degree Celsius.

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So, the third phase. Now, we are talking about the cooling phase this part of a phase. Here the aim is to cool the temperature to the room temperature again. The rate of cooling has to be in a phase that it does not have a thermal stress to the glass. We cannot have a rate at which we break the glass, because of cooling. And we cannot have a rate which is much slower which is not economically viable. So, normally it is in the same range of heating or just higher than the heating rate. So, cooling phase it ends normally at 70 degree Celsius since the Δe becomes Δt becomes very lower. It is impossible for us to cool further with the additional force. So, after 70 degree Celsius it is allowed and naturally to cool, but the faster rate of cooling is achieved between 280 to 70 degree Celsius.

So, typically if you have to put a cycle into this is a heating will be around 3 hours, and holding can be around 2 hours, and cooling roughly will be around 2 hours. So, a cycle of heat soak will have 7 hours. So, number of glasses depends on the oven type, number of tonnage of glasses in the oven depends on the oven type, and the heating capacity of the oven, in the calibrated structure of the oven, and our ability of load and unload the oven. So, this is what I am stressing upon the rate of cooling should be controlled to minimize the risk of glass breakage due to the thermal stress. If we cool faster rate, then there is a risk for breaking it. So, how does we do it?



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Heat soak process system

Consists of:

- ☒ Oven ;
- ☒ Glass support ;
- ☒ Separation system .

Note : The oven shall be calibrated, and this determines the method of operation of the heat soak process system during manufacture of heat soaked thermally toughened soda lime silicate safety glass.



The heat process heat soak oven normally consists of 3 things. One is oven for which we have a heaters, the second is a glass support, third is the separation system. The oven should be calibrated or shall be calibrated with this determines the amount at which the operation of the heat soak. The total processes depends on the calibration of the heat soaking.


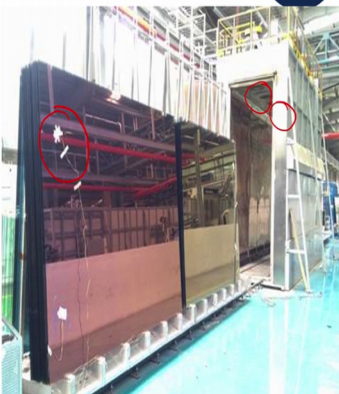

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Heat soak process system

☒ Oven

- ☒ The oven shall be heated by convection and shall allow an unhindered air circulation around each glass pane.
- ☒ In the event of glass breakage the airflow shall not be hindered. The airflow in the oven shall be led parallel to the glass surfaces.

NOTE The openings for the air ingress/egress should be designed to ensure that fragments of broken glass do not cause blockages.



So, this is the typical oven which we use here, here you can see glasses. So, area where I am marking is where the thermocouple is placed. So, here you can see the oven is made

of usually the ovens are heated by a conventional forces. It is not direct radiational it is more of conventional force, and it has to allow an unhindered air flow. So, you can see number of blowers at the top, and it has vents of the bottom to go out and keep recirculating hot air. And it is usually conducted heat flow one is working on that.

In case even there is a breakage if there is a breakage in the glass, we have to keep the separating systems and support systems such a way that it does not hinder the next glass. The aim in this process is to break the glass having the nickel sulfide. The aim is not to have glass error free glass circulated free glass. Aim is to have a defective glass broken in the oven. So, the key point is again highlighted here. The openings it has to be included to ensure that even the broken glass is there. It should not hinder the process of going it.

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Heat soak process system

Glass support

- Glasses may be supported vertically or horizontally. The glasses shall not be fixed or clamped, they have to be supported to allow free movement.

NOTE Vertically means true vertical or up to 15° either side of true vertical. The distance between glasses affects the airflow, heat exchange and the heating time. Glass to glass contact shall not be allowed.



The image shows a glass support system in an oven. The system consists of a metal frame with vertical supports. Red handwritten annotations include a box around the bottom support area and arrows pointing to it with the word 'Teflon' written in red. The top right corner of the slide features logos for NPTEL and GLASS ACADEMY.

The second surface is the glass support. Now, glass support here we are talking is about the vertical or the horizontal support. So, you can see the vertical support is and horizontal support is laid such a way that the glass should not be overly having additional stress in (Refer Time: 07:47). It is normally 15 degrees inclination in max of max 15 degree of inclination is allowed. And the glass has to be separated in such a way that it is supported uniformly.

So, here you can see several separators at the bottom, which is uniformly supporting. And the bottom support is always not with the direct metal contact. It has a Teflon which

abstract the contact between the oven. Remember you when you heating the furnace, when you are hitting the glass, even this steel structure will get heated. So, it cannot have a direct contact with the glass which will have an additional stress in to the local point, and it can cause a damage to this.

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Heat soak process system

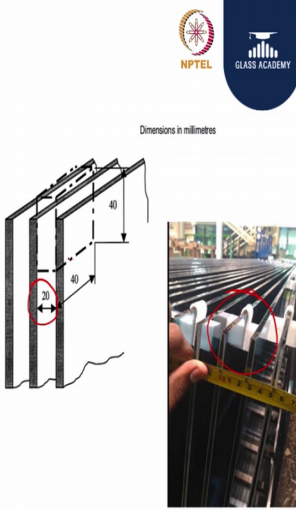
Glass separation

- ⊗ The glasses shall be separated in a manner that does not hinder the airflow. The separators shall also not hinder the airflow e.g. see Figure A.
- ⊗ The minimum separation of the glasses shall be determined during the calibration of the oven, see Figure A

⊗ **NOTE 1** Generally, a minimum separation of 20 mm is recommended

⊗ **NOTE 2** If glasses of very different size are put on the same stillage, they will require greater separation in order to prevent glass breakage when the furnace is opened after the heat soak process. The same applies to glasses with holes, notches and cut-outs.

Dimensions in millimetres



So, third part of it is the glass separator. You can see this is separators which are used. So, the aim is to have 20 mm evenly distributed over glass for example you have to have each glass the glass has to have a difference of 20 mm. And the distance of every 40 mm we need to have a separator. So, this all defined in this standards the where you have to create this spacer, how much is the space between the glass. And we normally recommend 20 mm is the space between the 2 glasses.

Here then key thing is, so ideally if the glass sizes are same, we do not have any differences, but when you have a glass say different glass put in the same stillage, it will require more separation. It does not fall only in the standards, because when you put more glasses, the first glass tend to stop lie on the next glass. So, we need to have more separators ensure that there is no physical contact between glasses, there is the uniform gap between glass at the area. And this does not have an additional stress introduced in to the next glass.

So, the care should be taken in terms of asymmetrical loading. Symmetrical loading is for easier than asymmetrical loading, but never the less this façade market does not

always have the symmetrical loading. So, in case of asymmetrical loading the care should be taken to have the spaces possible. So, the spacers are specifically designed for a numerous types of metal. These phases are designed to have whatever the size difference also. It can accommodate the asymmetrical loading.

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Calibration

- z The heat soak system, e.g. oven, glass separation, separators, etc., shall be calibrated
- z The calibration shall determine the heating phase of the process, glass separation distance, the positioning, material and shape of separators, the type and positioning of stillage(s) and define the operating conditions for use during manufacture.

Calibration criteria

- z The heat soak process system shall comply with the time/temperature regime as shown in Figure A.1.
- z The system shall be capable of meeting in the regime at both 100 % and 10 % load.

Key

T: Glass temperature at any point, °C	3: glass temperature
t: time, h	d: ambient temperature
t ₁ : time for the first glass to reach 280 °C	a: heating phase
t ₂ : time for the last glass to reach 280 °C	b: holding phase
1: first glass to reach 280 °C	c: cooling phase
2: last glass to reach 280 °C	

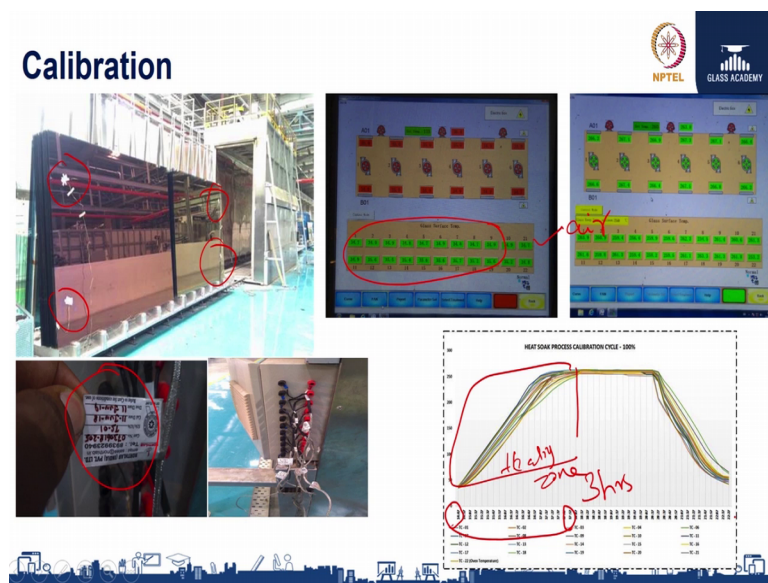
Figure A.1—Time-temperature regime as calibration criteria

So, the now I am coming to the key part that is the calibration of furnace. The heat soak system basically the glass when you calibrate, it is not only the oven when we calibrate it it is three things, which we calibrate the oven, the glass separation, and the separators all included we will calibrate it. So, this is for complete system the calibration for a complete system. So, how do we do it? We for example, if we have a 5 ton oven, normally we do for 100 percent loading and a 10 percent loading. So, we load the glass with 100 percent of glass 100 percent fully loaded and 10 percent. So, to operate normal operation will be around 40 to 50 percent of loading, so we do at extremes.

And we have 20 thermo couples put on the glass at specific locations. And the thermo couples are pre calibrated with the thermo couples are calibrated such a way that it does not have a differences of temper. So, it also has where which position which thermo couple is doing. And it will be monitored very closely with the systems which is having calibrated to each. It is done with that party normally there are the people of Hong Kong would do it the people from TUV they can do it.

So, you can see 3 in this graph you can see 1, 2, 3. So, 1 is first glass reaching the temperature, 2 is the last glass to glass is in the temperature, 3 is the glass which is reaching temperature, but should be maintained in such a way that it does not cross 320 degree Celsius. So, all three is important, one is there can be a first glass which can reach the temperature at t_1 ok. The last glass represent t_2 . And between this point there are classes which can go beyond 320. We should have a oven in such a manner that it can control that in the t_s temperature that should not go beyond t_{ok} . So, this graph is basically to explain it.

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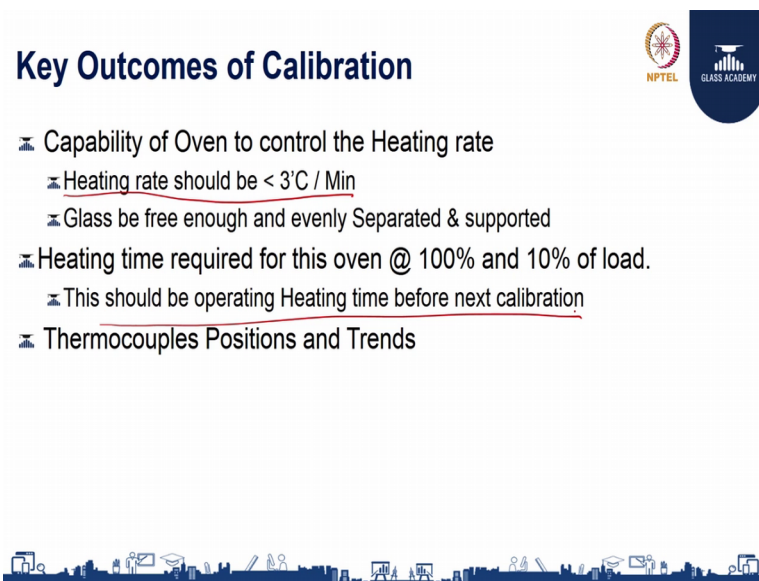


This is a how the typical process happens. So, you can see there are thermo couples laid at different locations. There are here 4 thermo couples like that there are back side there are 4 thermo couples. And inside the glass there are so 20 thermo couples will be there. So, these thermo couple has to be calibrated before we are putting into their operations. So, all thermo couples has to be connected in a proper way. At it has not should not have any bends and every things so that it can add an value in to it. So, it has to be properly connected. So, in a typical process you can see there will be 20 thermo couples plus 21 is the air temperature, we also measure the air temperature inside the furnace.

So, there will be 21 thermo couples which is measuring, so, this is start of glass you can see all thermo couples are 35 degree Celsius which is typical Indian temperature. And this is close to the end of the process where it is around 260 degree Celsius. And the air

temperature is also measured in all these cases. So, we wait until all the thermocouples reached down to 220 (Refer Time: 12:47), we get a graph close to this. So, here if you see this is the area, which is heating zone. So, if you have to mention the time, it is roughly 3 hours from here. So, 3 hours is taken for the heating, and then the holding is done for 2 hours after it is being cooled for almost 2 hours. This is the typical real calibration time one of the furnace of heat which we have.

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Key Outcomes of Calibration

- ▣ Capability of Oven to control the Heating rate
 - ▣ Heating rate should be $< 3^{\circ}\text{C} / \text{Min}$
 - ▣ Glass be free enough and evenly Separated & supported
- ▣ Heating time required for this oven @ 100% and 10% of load.
 - ▣ This should be operating Heating time before next calibration
- ▣ Thermocouples Positions and Trends

The slide includes the NPTEL logo and the Glass Academy logo in the top right corner. At the bottom, there is a decorative horizontal line with various icons representing educational and industrial themes.

So, key what is a outcomes we are looking on the capability of oven whenever we understand trying to understand the oven whether we are able to control the heating rate to less than 3 degree Celsius is the first thing which we are doing in the calibration. Is the glass free enough and evenly separated and supported. Glass should be free enough it has to be evenly supported and separated to all of the act to go through this. And the heating time required for this what we determine at the end of this is for 100 percent and 10 percent what is the heating time required for this. This is the operating time which we have to use until the next calibration is done. So, between normally it is done every year. So, between one calibration to other calibration the time taken to do the 100 percent and 10 percent loading, the is average time is used for using as a heating time. And thermo couple positions and trends which we understand from it.

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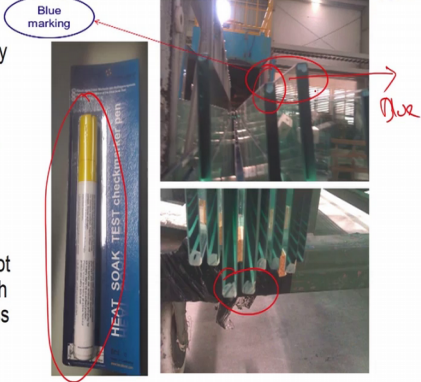
Heat Soaking Identification

Loaded Glasses

- Special marking will be done in any one edge of the glass (blue color dot)

Glasses unloaded onto trolley after Heat soak

- Blue marks will be changed to White color
- After Heat Soak, the Marking cannot be removed even if you scratch with your finger nails, (fused on the glass and permanent in nature)



The image contains two photographs of glasses in a furnace. The top photograph shows a blue marking on the edge of a glass, with a red circle and an arrow pointing to it. The bottom photograph shows the same glasses on a trolley, with the marking turned white, also circled in red. A red arrow points from the top photo to the bottom photo. A red circle highlights the marker pen on the left.

So, now we are going to the last part of the session where the heat soak. How do we identify the glasses are heat soak. We call we say we put it between the oven we take it out, and there are specific methods may be by which we can add it. So, for example in the top we can see a blue marking. So, this marking is done with a specific pen this is the heat soak co one marker pen. So, before putting in the furnace we have to mark it is a UV pen where we can mark the glass with a blue colour. And when it comes out of a furnace it comes in the grey colour. Normally it is done at thickness of the glass does so that it does not have any disturbance to the product quality.

And when you see a glass, which is heat soak will have this generally we will have this mark which means that it has gone through this oven. If it is not completed the cycle, this will remain as blue. So, we want this acetone from blue to white. And this is permanently fuse to the glass and it cannot be taken away by grinding or roughing of.

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Glass failure modes

Typical glass failure

- ⌘ A) Instability failure – compression member or flexural member
- ⌘ B) Overstressing of the glass in tension – by excessive uniform load, blast,
- ⌘ C) Impact, thermal stresses or uneven / inappropriate supports
- ⌘ D) Surface and edge defects
- ⌘ E) Solid inclusions

a) thermal failure b) hard body impact c) soft (spherical) body impact

d) hard spot on the edge e) inclusion

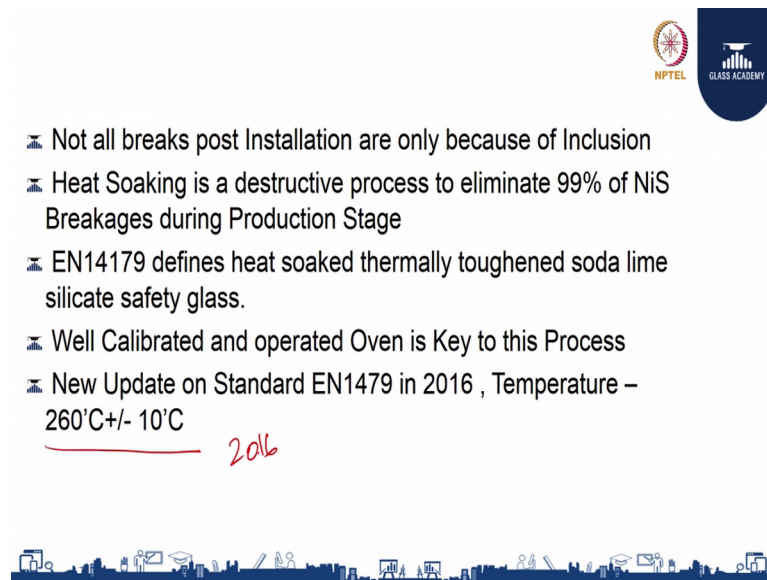
NO Butterfly pattern

NPTEL GLASS ACADEMY

So, now we understood what is heat soak and how it is used, and how it is calibrated, there are different patterns of glass. So, I summarize it what we have learnt. So, there can these kind of breakage is normally thermal breakage. So, when you see a breakage in the glass, it does not mean that it is always, because of nickel sulfide or because of any inclusion in the glass. We need to understand and observe closely the breakage pattern based on that we can conclude to a closest conclusion. For example, first breakage upon possible thermal instability breakage, here the compression is not done properly.

The second is and the over reason could be overstressing of glass in the tension, for example you can see a hard body impact with a hammer, with a stone everything can have a create an impact the breakage is typically like this. Third it could be soft body soft body which is spherical can be of small balls which has an impact the breakage pattern looks similar to make a sphere, but there is no butterfly. If it is a has some hot spot like a stone in during installation or a metal frame which is touching the glass which will create crackers like this. And in case of nickel sulfide breakages are inclusive breakages solid inclusion breakages it will have a breakage which is having a air lobe or a butterfly pattern.

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The slide features a white background with a blue border. In the top right corner, there are two logos: NPTEL (National Programme on Technology Enhanced Learning) and GLASS ACADEMY. The main content is a list of five bullet points, each preceded by a blue square icon. The text is in a black sans-serif font. The last bullet point, 'New Update on Standard EN1479 in 2016 , Temperature – 260°C+/- 10°C', has a red underline under '260°C+/- 10°C' and the handwritten number '2016' in red next to it. At the bottom of the slide, there is a decorative blue silhouette of a city skyline with various icons representing different industries and education.

- Not all breaks post Installation are only because of Inclusion
- Heat Soaking is a destructive process to eliminate 99% of NiS Breakages during Production Stage
- EN14179 defines heat soaked thermally toughened soda lime silicate safety glass.
- Well Calibrated and operated Oven is Key to this Process
- New Update on Standard EN1479 in 2016 , Temperature – 260°C+/- 10°C 2016

So, we are coming to end of this session. So, now couple of things have summarizes before I close. One is not all breaks post installation is because of only inclusions. Second thing is heat soaking is a destructive process which eliminate 99 percent of nickel sulfide breakages during production stage itself. EN 14179 is a standard which specifying the heat soaking and it will very detailed. And well calibrated and operated oven is the key to success of this process.

Now, the standard has been updated the temperature range has been changed from 280 plus minus 10 degrees to 260 plus minus 10 degrees based, this is done on 2016. This is based on several research works happened behind it to understand how the heat soaking can be made more effective, at least there are 100 to 150 journals which has been articles published on this heat soaking. So, this helps us to understand more on heat soaking.

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Summary:

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

- Heat soaking
 - Heating phase
 - Holding phase
 - Cooling phase
 - Heat soak process system - Oven, Glass support, Separation system
 - Calibration
 - Key outcomes of calibration
 - Heat soaking identification
- Glass failure modes
 - Instability failure
 - Overstressing of the glass in tension
 - Thermal stresses
 - Surface and edge defects

Thank you.