NICKEL SULPHIDE INCLUSIONS

A report on the phenomena of spontaneous breakage in Fully Tempered (Toughened) glass

by

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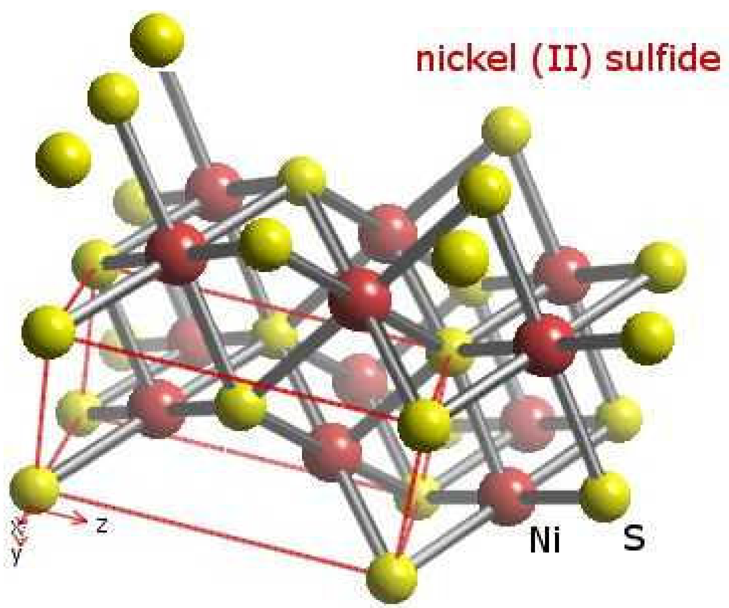

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PURPOSE OF THE REPORT

In countless cases where a spontaneous breakage of tempered glass has been attributed to nickel sulphide (NiS) inclusions, Architects, specifiers, building contractors and curtain walling sub-contractors often attempt to place the liability on the glass processor. Glass suppliers generally do not accept responsibility for replacement whether their warranties state the limitation or otherwise.

This publication shows that the wealth of information readily available on the subject makes this type of claim spurious if not vexatious and time wasting. Those involved in determining glass specifications can neither claim ignorance of the spontaneous breakage phenomenon nor shun responsibility for the consequences.

HEAT TREATED GLASS

To understand the content of this publication it may first be useful to first review the following appraisal of Heat-Treated glass. This technical bulletin from GGI provides a quick overview of the differing types of glass Heat Treatment with their physical properties and varying applications.

Additionally included are subject headings on; Annealed **glass, as this is occasionally misunderstood to be an alternative secondary heat treatment process and;** Spontaneous Breakage **as this figures largely in the purpose and development of certain heat treatments.**

WHAT TYPES OF HEAT TREATED GLASS ARE AVAILABLE?

1. **Fully Tempered (also known as Tempered or Toughened.)**
2. **Heat Strengthened**
3. **Heat Soak Tested**

Generally the first two glass types are made from Annealed float glass. Annealed float glass is ordinary glass, so called due to the nature of the manufacturing process.

ANNEALED GLASS

Annealed float glass is usually manufactured to ASTM C-1036 or BS 952.

In simple terms float glass is made by pouring molten glass onto a shallow bath of molten tin. Then drawing the subsequent 'ribbon' across the tin so that the flat surface of the liquid is duplicated on the two surfaces of the glass. As it is gently drawn from the pouring side of the tank it becomes cooler and solidifies. The glass surface condition being optically as flat as the liquid onto which it was poured.

The float glass cannot be immediately taken from the float tank as the outer surfaces of the glass will cool too rapidly and set up internal unwanted tensile stresses. It is therefore further processed through an Annealing kiln called a 'Lehr' to afford controlled cooling to near external ambient temperature and thereby remove all stress within the glass.

The resultant Annealed Float Glass can be further processed by cutting to size, drilling, beveling, polishing, brilliant cutting, et c.

The purpose of Heat Treatment therefore is to intentionally introduce a stress regime into the glass. The most common form being Tempered glass.

FULLY TEMPERED GLASS (ALSO KNOWN AS TEMPERED OR TOUGHENED)

What is Tempered Glass?

Tempered glass was originally developed as a safety glass. Primarily this is still what it is specified as.

How is Safety Glass Classified?

The classification of a safety glass is commonly expressed in international standards as one which, will either not break given a certain impact or break safely. The safe breaking characteristics are usually further described as not allowing large shards of glass that could cause piercing injuries.

How is Tempered Glass Made?

Tempered glass is made by heating the annealed glass to the softening temperature and then subjecting it to a rapid cooling with high-pressure air blowers. This rapid cooling affects the outer surface of the glass more quickly than the inner mid-plate and prevents it from returning to its original 'annealed float' molecular pattern. The process therefore deliberately sets up an internal tensile and opposing external compressive stress regime. The compressive strength targeted is designed specifically to yield the following characteristics: -

- **4-5 times greater resistance to mechanical forces.**
- **When broken shatters into small relatively harmless dice-like fragments.**

Why Stronger?

Annealed float is very strong in compression but very weak in tension.

Tempering glass induces high compressive strength on the outer surfaces of the glass. Subsequently the glass can sustain higher mechanical loading.

Why Safer?

If the tensile stressed inner mid-plate of the glass is compromised by a crack, the bond holding the glass together is broken and the entire panel will disintegrate into small dice. A person in contact with the glass stands little chance of sustaining a piercing injury.

Tempered glass Applications

- **Areas of glazing where accidental human impact is a possibility.**
- **Glazing that carries structural loads.**
- **Glazing that is expected to withstand high wind loading.**

- **Glass installations where thermal shock risk is present.**

HEAT STRENGTHENED GLASS

What is Heat Strengthened Glass?

Heat Strengthened glass is glass that is stronger than annealed glass by a factor of approximately 2 to resist mechanical load, but is not a safety glass.

How is Heat Strengthened Glass made?

The product has undergone a similar heat treatment process as tempered glass using the same plant and equipment, with two exceptions. The differences are that the heating cycles are slightly different, and rapid cooling is not with such high pressures.

Why have Heat Strengthened glass?

Heat strengthened glass was developed to tackle a very specific phenomena that infrequently occurs with Tempered glass. The effect of total disintegration of tempered glass when broken for no apparent reason can be dramatic when it happens. Spontaneous breakage in tempered glass, although rare, commonly arouses strong and mixed reactions from client and contractor alike. Heat Strengthened glass is unlikely to suffer the phenomena and does not disintegrate in such a spectacular fashion. This is because the Heat strengthened glass breakage pattern is very similar to Annealed float glass.

A further benefit to utilising Heat Strengthened glass is that the 'roller wave' surface distortion produced in the Heat Treatment process is less pronounced than that for Tempered.

Heat Strengthened Glass Applications

- **Glazing that is expected to withstand high wind loading.**
- **Glass installations where thermal shock risk is present.**
- **Glazing where roller wave requires minimising.**
- **Glazing where spontaneous breakage is to be avoided.**

HEAT SOAK TESTED GLASS

What is Heat Soak Tested Glass?

Heat Soak Tested or Heat Soaked Glass is a Tempered glass that has undergone additional heat treatment intended to eliminate by destruction any glass panels that have a characteristic that may latently manifest as a spontaneous breakage after installation.

What is the Heat Soak Test?

After tempering the glass is batched and stacked in a static oven. The temperature is held at a high enough level to induce accelerated conditioning but not so high as to affect the desired stress regime in the glass.

The required conditioning is for three purposes: -

1. **To increase the growth of any microscopic vents from the glass edge or surface sufficient to create a compromise of the tensile mid-plate and cause a subsequent glass breakage.**

2. **To increase the growth of any microscopic vents around the surface of a microscopic foreign body within the glass and thereby cause a spontaneous breakage.**

3. **To increase the growth rate of any Nickel Sulphide inclusions setting up a chain reaction that may also cause a subsequent breakage.**

Heat soak testing is expected to eliminate 98% of those tempered glass panels from production that may have otherwise sustained a spontaneous breakage.

SPONTANEOUS BREAKAGE

How Does a Spontaneous Breakage Happen?

Spontaneous breakage can occur for several reasons. The most common being edge defects, caused either during processing or installation; Surface damage, caused either before or after installation leaving deep scratches or pitting and foreign bodies such as nickel sulphide in the glass from which, vents (cracks) can initiate or grow.

Why should Nickel Sulphide Cause a Spontaneous Breakage?

Nickel Sulphide is only found in very minor quantities during a typical year of production from a float line. It has always been present in glass but can only cause the highly emotive spontaneous breakage in tempered glass.

Nickel sulphide inclusions are mostly so small that they cannot be detected by any commercially viable method. Nonetheless they are capable of causing breakage by virtue of the necessity to change shape and volume in tempered glass where only a small vent is needed to cause breakage.
During the tempering heating cycle the glass is heated to around 650° C. At over 450° C a nickel sulphide (NiS) inclusion will change shape with an accompanying volumetric reduction. Whilst the glass enters the 'quench' phase of tempering the NiS is 'frozen' in an unnatural state for the ambient temperature. Over time, with weather heating cycles the NiS will attempt to revert to its' stable shape. Add to this wind developed membrane stresses, and this frequently results in the manifestation of a spontaneous breakage.

AVAILABILITY OF INFORMATION

1962 **"Fracture of Toughened Glass Wall Cladding, ICI House Melbourne" Report by E. R. Ballantyne of the CSIRO (Commonwealth Scientific and Industrial Research Organisation)**

This report was the first to show the cause of previously unexplained breakage to be due to NiS inclusions.

1964 **In the bulletin of Anglo-Japanese Economic Institute in May 1964 the General Agents for Glaverbel SA in Australia published a letter explaining that the glass involved in failures due to NiS on ICI house in Melbourne was of German origin and not, as had been reported in the press, Belgian.**

1966 **N.I. Amosov, of the Skeklo Trudy Institute in Stekla published "Spontaneous Cracking of Hardened Glass"**

1969 **Pilkingtons introduced Heat Soaking as part of the normal production process for Armourclad glasses.**

1970 **Pittsburgh Plate Glass (PPG), Pennsylvania, issued to all their customers a four page official publication entitled "Heat Strengthened Spandrelite", (a cladding product of theirs) which claimed, among other things, that** ***"No unexplainable breakage has occurred in PPG Spandrelite"***
and
'Spontaneous breakage attributable to occlusions has not occurred in PPG's Spandrelite"
and
"To avoid 'spontaneous' breakage, which has been experienced on some spandrel glass installations, we recommend that the maximum as well as the minimum values of surface and edge compression be made a performance requirement as follows: -
Surface compression range 3,500 – 7,000 PSI
Edge compression range 5,500 – 8,500 PSI

1972 **Bauwelt, 23rd October (A West German Architectural Journal), an article discussed NiS and Heat Soaking in simple terms for the building industry generally.**

1973 **I. Merker (Flachglas), gave a lecture to the German Flat Glass Technology Group meeting, later published in "Glastechnische Gerichte Vol. 47, Part 6, 116 –121.**

1974 **The UK Dept. of the Environment, Property Services Agency, issued in October of this year their "Technical Instruction No. 63. Breakage of 'Armourclad' Toughened Glass". This dealt with the risk of injury to the public from breakage of Armourclad when used for external wall cladding** ***"Where breakage could cause injury to people its use is prohibited pending the***

completion by the manufacturer of tests on new treatments and suitable glazing techniques"

From about 1976 onwards, more popular articles about 'spontaneous breakage' began appearing in the trade press, particularly in the USA, and the industry as a whole knew at least something of the story, as did many of the major Architectural Practices. That is, the story was spreading outside the Glass Industry and into the Building Industry generally.

1977 **C. C. Hsiao, (University of Minnesota, Minneapolis, Minnesota) gave a paper entitled "Spontaneous Fracture of Tempered Glass"at an international conference on fracture at the University of Ontario. This was subsequently published in 1978 and was perhaps one of the more accurate full technical discussions, in English and in easily accessible form.**

1977 **Richard Wagner, of Saint Gobain Industrie published a paper in Glastechn. Ber 50. Nr. 11. 296-300 titled "Inclusions de Sulfure de Nickel dans le Verre". This described the problem and gave a tentative explanation of the mechanism by which the particles are formed.**

1978 **Schott, K. and Koldehof, W. published an article, "Cladding panels made from glass – Applications and insurance" (In German) in Glaswelt. This was summarised as –** ***'The use of Glass for external applications in building is discussed with particular reference to the effect of nickel sulphide inclusions'***

1979 **L. Jacob, of Jacob and Associates, Australia, presented a paper at the International Conference on Materials, Cambridge – Vol 3., Pergamon Press, entitled, 'A Fracture Mechanics Approach to the Determination of the Failure Mechanism for Heat Treated Glass from Nickel Sulphide Inclusions and Surface Degradation (Scratches)'**

By 1980 knowledge of the problem was spreading more widely and some independent glass tougheners were beginning to offer heat soaked versions of toughened glass for when it was to be used in 'critical' locations.

1980 **'A Fracture Mechanics Description Of The Microcracking About NiS Inclusions In Glass' by M. V. Swain was published in the journal of Non-Crystalline Solids, (an American Journal) by North Holland Publishing Co. It identified the composition and size of NiS inclusions.**

1980 **Libbey Owens Ford (LOF), the leading primary glassmakers in the USA published a Technical Information paper in America. It described fully tempered and heat soaked glass, and discussed NiS inclusions. LOF stated that all fully tempered glass supplied by themselves for skylights, sloping glass and spandrels would be heat soaked.**

1981 **The Journal of Materials Science, 16(1981) 151-158, carried an item, "Nickel sulphide inclusions in glass: an example of microcracking induced by volumetric expanding phase change" by M. V. Swain, of Saint Gobain**

Recherche, Aubervilliers, France. It covered the description of the phenomena generally and went on to develop a ***'simple generalized fracture mechanical analysis of the cracking about such inclusions. The critical dimensions of the inclusion to initiate microcracking in an annealed specimen and to cause spontaneous fracture of a tempered plate are predicted'***

1982 A paper by Werner Klein, published by Schott, the German glass producer, stated that toughening glass could induce the risk of failure as a result of NiS inclusion expansion. He said that the risk was low and quantified it as about 1%.

1983 The American AAMA (American Aluminium Manufacturers Association) published 'Sloped Glazing Guidelines'. In the section on tempered glass it said: ***"Heat soaking of tempered glass is performed by some manufacturers to reduce the frequency of inclusion (small foreign particle) related breakage"***

1985 Viracon, in their 'Tech Talk' newsheet, dealt with NiS inclusions and the effect of heat soaking quite thoroughly, but concluded that to eliminate all inclusions the length of the heat soak should be 5 to 8 times longer that was currently being used by most manufacturers.

1986 Pilkington Product Literature published for their Armourplate Glass Spandrels for Curtain Walling, claimed heat soaking as standard. However, it was not until 1989 before new product literature stated that the product would be heat soaked for roof glazing, balustrades, and spandrel panels or when specifically requested. They also offered heat strengthened glass at this time.

1988 'Suppliers Recommended Limiting Use of Tempered Glass in Buildings', an article by John Swanson was published in the American magazine Glass Digest. The article stated that PPG would knowingly supply tempered glass only for a limited number of defined usages. The reasons given were that ***"even though it is a very, very small percentage, there's some concern about nickel sulphide inclusions in tempered glass"***

1988 PPG published in their Technical Service Recommendations a guide called 'Use PPG HESTRON Heat Strengthened Glass for Architectural Glazing'. This was updated in 1992. They state in this publication:
"PPG does not recommend the use of fully tempered glass for the overall spandrel and vision areas of high-rise commercial buildings for the reason that the original float glass as made by any manufacturer may contain some allowable imperfections of a certain size and character (e.g., nickel sulphide stones) that may cause such glass to spontaneously break while in place above ground level on the building."
and
"PPG's heat strengthened glass is almost never subject to spontaneous breakage due to nickel sulphide stones because of carefully controlled, limited upper levels of surface compression and resultant balanced centre tension stresses."
and
"Heat soaking of fully tempered glass is a significant waste of energy, is not

completely effective and, therefore, provides little additional information for estimating the probability of breakage caused by nickel sulphide stone inclusions in fully tempered glass which has been heat soaked."

1990 **The UK Glass and Glazing Federation (GGF) published the UK's first independent data sheet on the subject of spontaneous breakage contained within their section on Heat Treated glasses of the GGF Glazing Manual. This Manual is the handbook for the Glass Industry and is referred to Countrywide from Government authorities and architectural practices to small glazing Companies.**

1990 **The German Standards Organisation DIN published DIN 18516, part 4 for 'Back Ventilated, Non-Loadbearing, External Enclosures of Buildings, made from Tempered Safety Glass Panels'. Essentially 'spandrel' panels. Section 2.5.1 states a requirement for an Elevated Temperature Test. Although the reason is not identified, this is clearly a Heat Soak Test. The limited volume of text leaves scope for ambiguity. Nonetheless this represents the earliest attempt by a standards organisation to recognise a need to set boundary conditions for the Heat Soak Test. The exact section translation reads:**
"2.5.1 Elevated Temperature Test
The manufacturer shall condition all panels of one consignment at a mean oven temperature of (290 ±10)ºC for eight hours and shall issue a DIN 50049-2.1 or a DIN 50049-2.2 Inspection document covering such testing."

1990 **The Flat Glass Marketing Association (FGMA) of the USA also published in their 1990 edition Glazing Manual details concerning spontaneous breakage. The advantages of using heat strengthened glass over fully tempered are discussed and the Manual states:**
"There is no known method to inspect for these virtually invisible inclusions or to eliminate them from the glass batch."
and
"Not all technologists agree that heat soaking will significantly reduce the risk of glass breakage from nickel sulphide stone inclusions. Additionally, the process adds a significant cost to the tempered glass product."

1992 **ASTM C 1048 – 92 is published giving guidelines to glass processors for the testing and assessment of Heat Treated glasses closely defining the surface and edge compressive stress levels associated with Fully Tempered and Heat Strengthened glass. This was updated with new parameters in 1997.**

1994 **The Glass and Glazing Federation (GGF) published section 7.1 of the Glazing Manual entitled ' Non-Vertical Overhead Glazing: Guide to the Selection of Glass from the Point of View of Safety'. This guide being primarily concerned with the fall out characteristics of safety glass states under the description of toughened glass that** ***"Thermally toughened glass may contain nickel sulphide inclusions which could cause spontaneous breakage, although the risk of this happening is very low"***

In the same data sheet a separate section describing Heat Soaked Toughened Glass states *"Heat soaked toughened glass has the same properties and breakage characteristics as thermally toughened glass. However, the risk of spontaneous breakage due to nickel sulphide inclusions is negligible."*

1995 **British Standards Institute published their first reprint of BS 952 : Part 1, Glass for Glazing, Classification since 1978. This standard is referred to in almost every glass specification preamble. In section 4 of this standard definitions are provided for 'Thermally Toughened Glass', 'heat Strengthened Glass' and 'Heat Soaked Thermally Toughened Glass'. The latter is described as *"thermally toughened glass which as part of the manufacture has been subjected to a sustained temperature for a specific time such that a large proportion of any nickel sulphide inclusions present are converted to the stable form"***

1997 **The Glass Association of North America publish the revised Glazing Manual formally published by the Flat Glass Marketing Association (FGMA). In this update spontaneous breakage is specifically discussed with a conclusion made that Heat Strengthened glass is the preferred choice of product to control the problem of NiS induced breakage. They state *"The potential risk associated with nickel sulphide inclusions can be minimized by using heat-strengthened glass."***

1997 **The Draft European Standard prEN 1279, Glass in Building- Insulating Glass Units notes the usual list of standards for further reference. All 'pr' prefixed Standard Numbers for Glass in Building Standards are under the auspices of CEN/TC 129. One such standard referenced therein is *"prEN: Glass in Building- Heat Soaked Thermally Toughened Glass"***
Manufacturers and specifiers in the European arena rapidly adopted the standard prEN 1279 through the latter part of the 1990's.
Many specifications around the beginning of the year 2000 required Insulating Glass Units to have CEN marking.

From the early 1990's all major producers of float glass carried information about nickel sulphide inclusions and the associated breakage in tempered glass and offered Heat Strengthened glass. Some additionally carried statements about Heat Soak Testing. Then however, there was still no consensus on the usefulness of Heat Soak Testing or its parameters.

The following glass manufacturers carried details on the subject in their technical literature.

- **Pilkington**
- **Glaverbel (including Asahi, Japan.)**
- **AFG**
- **PPG**
- **Guardian**
- **Visteon (nee Ford)**
- **Viracon (although not a manufacturer Viracon are a major glass processor)**
- **Interpane**

- **Saint Gobain**

In addition Trade Associations and Standards Organizations have frequently during the 90's made reference in their published literature to NiS.

2000 **The Institute of Structural Engineers (UK) in December this year publish a handbook on STRUCTURAL USE OF GLASS IN BUILDING. This comprehensive guide includes a section on Point-Supported glass where the use of toughened glass is explained as almost always essential. The paragraph on toughened glass states.**

"It should also be heat-soaked as a quality check, to reduce the incidence of nickel sulphide inclusions in panels leaving the factory that would otherwise cause spontaneous fracture of the glass"

2000 **CEN/TC 129 published the draft document for Glass in Building – Heat Soaked Thermally Toughened Soda Lime Silicate Glass, part 1 : Definition and Description. At this time the provisional standard did not have an allocated number.**

2000 **An article was published in the May issue of the UK Institute of Building, Construction Manager magazine concerning a panel of glass falling out of the Millennium Product Award winning Neathouse Place office development opposite Victoria Station, London. A spokesperson for the owner in referring to the main contractor, Wates, is reported as saying**

"Wates has to put the building in good order. While it is early days, we suspect that the glass shattered because of Nickel Sulphide inclusion."

The article continues

"Buro Happold. The engineer for the refurbishment contract, has been called to investigate, she said. Westminster Council is also investigating the accident. The incident follows a similar case six months ago when two other panes shattered and fell out of their frames at Neathouse Place ----"

This single building itself would be enough to highlight the level of knowledge permeating outside of the Glass industry concerning NiS breakage. Just for good measure however the article draws attention to spontaneous breakage in glass by referring to what it describes as a "spate" of shattering glass incidents.

The article concludes with the following statement.

"In February, the Health and Safety Executive was called to investigate shattering glass at Stratford and Canary Wharf Jubilee Line underground stations. Other buildings suffering from glazing failures include the £120m Eurostar terminal at Waterloo, Richard Roger Partnership's £40m law courts in Bordeaux and the new National Glass Centre in Sunderland."

2000 **EN 1863 –1 : 2000 GLASS IN BUILDING – HEAT STRENGTHENED SODA LIME SILICATE GLASS.**

Published in January the first European standard for Heat Strengthened glass clearly marks recognition for the increasing demand for the heat treatment that obviates Heat Soak Testing.

2001. **May 2001 Draft European Standard prEN 14179-2 was published the standard covers: GLASS IN BUILDING- HEAT SOAKED THERMALLY TOUGHENED SODA LIME SILICATE SAFTEY GLASS- PART 2: EVALUATION OF CONFORMITY.**

The draft document outlines procedures for testing Heat Soak Glass to maintain compliance with standards relating to safety and security classifications. Additionally outlined are procedures for voluntary marking or document control mechanisms. As at 18th July 2003 Technical Committee CEN/TC 129 classified this standard as still 'under development'.

2001. **June/July 2001. G. James, Australia's leading glass and aluminium processor and contractor published a 122 page Glass Handbook. Widely distributed, the book was sought after by many Australians working in architectural glass and associated fields. Section 4 on Heat-Treated glass accurately describes the industry position on spontaneous breakage and advocates Heat Soak testing specifically**

" …for toughened glass assemblies or where the consequence of breakage could result in injury"

A strong recommendation that promotes considered and economical specification of the Heat Soak test.

The beginning of the new millenium saw a huge increase in the Heat Soak test being specified across the world. Many specifiers however resort to the German standard perhaps in the mis-guided belief that 8-hours is better than 2, by not delving into the parameters set by the more effectively controlled 2-hour test. In addition, the paranoia that surrounds the mystery of glass breaking without being touched has condemned many clients to pay exorbitant capital costs for buildings with all of the toughened glass heat soak tested.

2001. **December 2001 Draft European Standard prEN 14179-1 was published the standard covers: GLASS IN BUILDING- HEAT SOAKED THERMALLY TOUGHENED SODA LIME SILICATE SAFTEY GLASS- PART 1: DEFINITION AND DESCRIPTION. The new standard clearly defines boundary conditions for the Heat Soak Test including : glass temperatures; oven temperatures; glass stacking system; heating phase; holding phase; cooling phase. Importantly the holding time is stated as being 2 hours in section 5.3.3.**

"5.3.3 Holding phase

The holding phase commences when the surface temperature of all the glasses has reached a temperature of 280ºC. The duration of the holding time is 2 hours”.

As at 17th July 2003 this Draft was classified by Technical Committee CEN/TC 129 as ‘under approval’.

CONCLUSION

The above list is by no means comprehensive but simply serves to reinforce the statement made at the start of this document that those in the business of either specifying glass or installing it will be considered aware of both spontaneous breakage and the association with NiS.

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