Nickel Sulphide Inclusions - Important Issues for the Designer

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Abstract

Nickel Sulphide induced spontaneous failure of toughened glass continues to occur in a few prestigious buildings. Unfortunately, there is no immediate solution to eliminating this problem. Heat soaking techniques are being formulated to minimize the risk of spontaneous fracture in toughened glass.

This paper focuses on the current position and the issues available to designers. As well as discussing the key issues, methods of predicting further breakage are presented, so that, based on the known success rates of heat soaking, confidence can be gained in using the product when the risks are taken into account.

Introduction

Despite the reported efforts of manufacturers to eliminate or reduce the problem, Nickel Sulphide (NiS) induced failure in toughened (fully tempered) glass used in buildings continues to occur around the world. Everyone involved is aware of several projects, where such failures have occurred in the past few years. Litigation as a consequence of NiS induced failures in prestigious buildings has made the industry aware of the problem. The associated costs of litigation is a serious matter and is of concern to all involved with the use of toughened glass in buildings. Furthermore, the risk of injury to the public as a consequence of NiS induced failures in toughened glass is a constant threat to building owners. The problem is not restricted to anyone particular manufacturer and is global. Consequently, this remains a real problem for all the designers as to the appropriate action required in order to minimise this risk of failures, subsequent litigation and potential injury to the public when toughened glass is required to be used for strength reasons.

The primary options available to the designers in the past have been to specify glass free of NiS inclusions, Heat Strengthened glass with a reduced level of surface compression or Heat Soak testing. However, none of these types of specifications are entirely suitable, as we are not aware of any manufacturer that can guarantee NiS free glass, or give guarantees that such breakages will not occur in Heat Soak Tested glass or in Heat Strengthened glass.

The objective of this paper is to review and present the issues, as they currently exist, from the perspective of the designer.

Current Position

The designer is confronted with a difficult option as to his position with reference to the specification, selection and use of toughened glass in his/her building. Currently there exists only one standard (German Standard DIN 18516, 1993), which makes reference to heat soaking as a potential solution to the minimisation of NiS induced failures and toughened glass. The current prEN 14179 draft standard is expected to be published later this year. This standard is the most comprehensive approach yet made to provide a test paradigm for the elimination and or the minimisation of NiS induced failures. The DIN 18516 standard has been shown to be inadequate and the prEN 14179 standard is yet to be internationally accepted and validated.

Some of the key issues related to the requirements and the criteria identified in the DIN 18516 standard and the prEN 14179 draft standard is presented below. A review of the criteria in these two standards raises important questions that will eventually need to be clarified before the glass industry has a truly acceptable heat soak methodology which will potentially minimise and ultimately eliminate NiS induced failures in toughened glass.

The key factors of the prEN 14179 draft standard are summarised as follows:

- Optimum temperature - 290^{0}C ± 10^{0}C
- Hold time – 2 hours
- No heating or cooling time specified
- All glass must reach a minimum of 280^{0}C
- The heat-soaking oven must be calibrated.

These five factors bear consideration as follows. The existing DIN 18516 standard also requires a heat soaking operating temperature of 290^{0}C ± 10^{0}C, but the difference in the specifications between the DIN 18516 and the prEN 14179 is that the prEN 14179 requires that all glass needs to reach a minimum temperature of 280 ^{0}C before the commencement of the hold for a period of two hours.

The heating and cooling periods in the prEN 14179 draft standard are not defined. This is of serious concern as the criteria in the prEN 14179 states that 6 or 8 mm glass may be used for the original calibration of the heat soaking oven. When other glass is used it would appear that the prEN 14179 standard relies on the minimum specified spacing between glass panels to ensure that the specified glass temperature is reached before commencement of the hold period. However, the theoretical consideration of heat transfer indicates that the time required to heat a panel of 19 mm glass (although less than 10 minutes) is almost 10 times longer than that required to heat a 6 mm thick panel of an equivalent size, so the effective holding period for the thicker glass would be less than the 2 hours specified.

Furthermore, for a given volume of heat soak oven, the mass of 19 mm glass would be more than twice the mass of 6 mm glass having the same gap between panels. Thus, the energy required to reach the same temperature would be more than doubled and therefore the calibration procedure currently specified may not be adequate.

Nevertheless, the calibration of the oven at 10 percent and 100 percent load capacity is the only long-term criteria used in the prEN 14179 draft standard to ensure that all future batches of glass will achieve the critical temperature before the commencement of the hold period in the heat soaking process. This is a matter of concern as mixed batches of glass will require different periods of time for the basic heating of the panel to the minimum 280^{0}C before the commencement of the hold period.

Even if a specific oven is correctly calibrated for a 10 percent and 100 percent load capacity and then if the glass temperature is not reached because of the size of the batch or the thickness of the glass used then be manufacturer or processor would be reliant on luck rather than good practice before the commencement of the hold period to ensure that the transformation does actually take place. The concern here is the quality of the calibration and its reliability for the total process. Once the calibration is completed then there is no other mechanism or procedure that the processor can depend on to ensure that the glass temperature is reached before the commencement of hold period and their confidence as to the adequacy of the heat soaking process is potentially in doubt.

The worlds leading glass manufacturers still have different opinions in respect to the adequacy and reliability of the heat soaking regime.

Jacob [1] has observed that some major glass manufacturers do not recommend the use of heat soaking as they consider it both expensive and unreliable in eliminating all NiS inclusions in the batch of glass. Other manufacturers use Heat soaking as a tool to sell their product and offer warranties to the quality of their heat soaking. For instance it is common to be offered a guarantee that a particular heat-soaking regime will provide a 95 % confidence level of eliminating all the critical NiS inclusions. Unfortunately, it is difficult to understand what this confidence level exactly means. In discussions with numerous companies that offer heat soaking, one may get different explanations as to what their individual guarantee means.

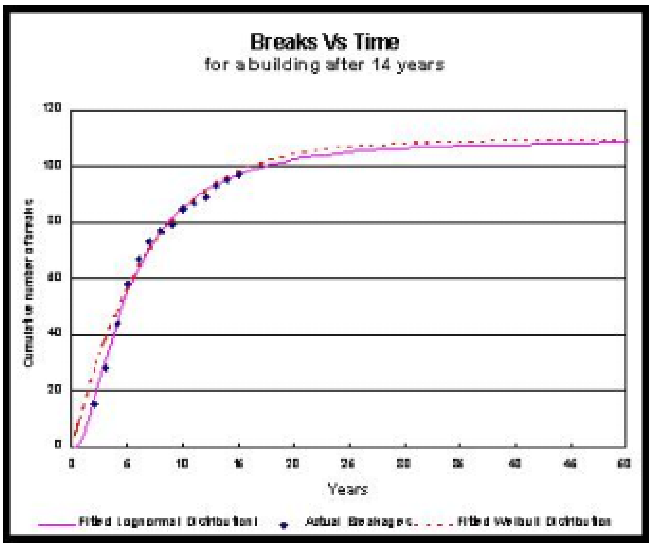
Success rates for heat soaking

It is generally commercially considered that the success rate of heat soaking may be taken as being a confidence level of 95% (or 98.5 % for other regimes). For the 95% confidence level does it mean that 95% of the glass that has been subjected to the heat soaking is free of Nick Sulphide and consequently there could be 5 percent of the panels with NiS left in the batch. Or does it mean that 95% of the panels with NiS inclusions have been eliminated leaving only 5 percent of that the number of panels found with inclusions still left in the batch? The designer is not likely to be made aware of the number of failures that had occurred during the heat soak process. So the designer or the building owner will be unable to quantify the number of failures that can be expected on the building. Furthermore, the quantity is relative to the amount present in the batch and this varies from batch to batch. Consequently, even when the heat soaking results are made known, then what are the acceptance criteria to be used?

It is important to recognise that the heat soaking process is fundamentally very effective. The problem is what is the correct temperature and time regimes that will provide the best results for the customer?

The prEN 14179 recommends 290^0 C $\pm 10^0$ C is this correct? Bishop et al [10] has demonstrated that the transformation temperature for NiS in the beta phase can vary as a function of the time and the composition of

Fig 1



the inclusion. It is well recognised that NiS inclusions come in various forms and various compositions [2&3]. This must be reflected in the heat soaking paradigms. Furthermore the presence of other impurities such as Iron has been shown to have significant influence and impact on the transformation time of NiS. This factor is not adequately reflected in the heat soaking paradigm as defined by prEN 14179.

Predictive Techniques

The current thinking is that the phase change mechanism is one of the primary causes of spontaneous failure in toughened glass. Other factors like the level of toughening, membrane, bending and thermal stresses, and purity of the NiS inclusion are important issues that will influence the fracture rate of the toughened panel.

Research being undertaken by major glass manufacturers is reportedly focusing only on the phase change mechanism [3&4]. Their approach must be applauded. However, it must be recognized that heat soaking does not completely eliminate NiS induced failures in toughened glass. Consequently there is a need to define an acceptable level of NiS induced failure after heat soaking.

Review of some major buildings that the authors have been involved with have also shown that failure of toughened glass could result from mechanical damage to the glass surface/ edges. Unfortunately the fracture pattern from mechanical damage can also be mistaken to be NiS induced failures when the fracture origin is lost. Exposed edges of structurally glazed panels may get damaged. These edges are subject to some level of stress, dependant on the magnitude of the damage and the level of wind loading, the induced stress can cause crack growth. This crack growth can and will cause spontaneous fracture. This type of failure may incorrectly be attributed to NiS inclusions.

Discussions with people involved in the development of the prEN 14179 have revealed that the failures during the heat soaking process were determined using microphones to signal the time at which each failure occurred. The fracture origins were not located and examined and thus they did not eliminate the failures attributable to thermal shock during the heating and possibly cooling process of the heat soaking. This obviously distorts the statistics that have been used in the development of the heat soaking process. Furthermore, it will also distort the statistics on which the 95% confidence is based.

Kasper [3] has reported a good correlation between numbers of failures and the Weibull distribution. Jacob [5] has also demonstrated this correlation and he used the Weibull distribution to predict future failures on a specific project. Calderone has also shown that the Weibull distribution is a versatile model that may be used but proposed that the lognormal distribution is better for predicting the total number of NiS induced failures in toughened glass. This is due to the Weibull distribution being simply a mathematical model while the lognormal distribution is a fundamental distribution, which occur when the variate is the product of many independent and random variables [6]. The time to failure due to NiS inclusions is affected by many independent variables. Such variables include: the size of the NiS inclusion, the location of the inclusion within the glass, the stress levels to which the glass has been tempered, the temperature history to which the glass has been subjected and the composition of the inclusion. Thus, it is not surprising that the lognormal distribution has been found to fit the glass breakage data very well (see figure 1).

Concentration of Nickel Sulphide in glass

Various researchers have attempted to define the concentration of NiS in batches of glass. These estimates have ranged from one inclusion for every four tonnes [7] to 1 inclusion in every six tonnes [8]. Pilkington's have stated that in the Seventies they found concentration of one inclusion in every 500 kilograms, while Kasper has reported batches with concentrations as low as 1 in 38.5 tonnes of raw glass [8].

There is no definitive and proven concentration of NiS in any glass, so further monitoring will be necessary before this statistics can be quantified. It would be ideal to have a worst case scenario of concentration, which will help establish the success or failure rate of any new heat soak regime.

Heat Strengthened Glass

Heat strengthened glass was developed to solve two problems;

- Thermal stress fracture
- NiS induced failures.

However, there have been numerous examples of installed heat strengthened glass fracturing spontaneously due to the presence of NiS inclusions.

The industries improved understanding of the impact of the induced tensile stresses in tempered glass has brought about a gradual decrease in the permissible level of surface compression in heat-strengthened glass. This is a positive step. There is now available suitable equipment to accurately measure the level of surface compression in heat strengthened glass.

The problem is the cost implications in having to heat strengthen glass to a very tight surface compression range. This permissible surface compression range is gradually narrowing. We have recently read a specification where the permissible surface compression range was specified to be between 35MPa and 45MPa. This is not a practical requirement or specification unless the customer is prepared to pay the extra cost.

Fig 2

Fig 3

Heat Soaking – further considerations

Is heat soaking an acceptable option? To those that are in a position to offer heat soaked toughened glass the answer will be yes. To those companies that supply tempered glass and do not have the facility to heat soak the answer will be no.

Jacob [1] identified various factors that affect whether or not heat soaking is successful, such as:

- Purity of the inclusion
- Size of the inclusion
- Location of the inclusion
- Level of tension within the tempered glass

One important factor in the success of the heat soaking process is weather or not there is a vent associated with the inclusion. If there is no vent associated with the inclusion then the heat soaking process will not cause the tempered glass panel to fail unless the volumetric expansion (due to the size and composition of the inclusion) is sufficient to cause a new vent to be generated. Furthermore, if the stone is very impure then the heat soaking process will not cause failure in the oven. However, this impure stone can cause failure in the installed panel at a later date. This is because of other factors such as membrane, bending or thermal stresses and crack growth acting on the vent associated with the inclusion. An example of a fracture origin from such a case is illustrated in figure 2.

There have been instances where toughened glass had been through the heat soaking process on numerous occasions without failure in the oven. Also, extremely small NiS inclusions can become a problem after heat soaking because of the associated crack growth with the application of additional stress.

Results of recent investigations

A recent study of NiS induced failure in a building showed that toughened glass heat soaked to the DIN 18516 standard had only achieved a conversion rate of 74%. Figure 3 shows the inclusion from the fracture origin of one of these panels, which was not eliminated in the heat soaking.

The replacement panels for the project were then heat soaked to the requirements of the prEN 14179 draft standard of 2 hours of hold time with an additional regime of holding at a temperature below 282^{0}C for 1 hour prior to the 2 hours at 290^{0}C. The lower (282^{0}C) value was selected, as it is the transformation temperature of NiS for sulphur-rich inclusions, as seen in the phase diagram given in figure 4. The result was that 6 breakages occurred in a total of 18 tonnes of glass heat soaked. Was this a bad batch of glass or was the heat soaking very successful and in the 95% confidence range? It was noted that all the failures occurred within the first hour of holding. Does this mean that the higher soaking temperature is ineffective or unnecessary?

In a similar study of another building with NiS induced failures a conversion rate of less than 70 % was found. This could be attributed to the higher temperatures in the heat soaking process.

Anecdotal evidence from numerous companies involved with heat soaking indicates that most of the observed failures occur during the first hour of the heating phase of the heat soaking cycle. This confirms our observation that the critical temperature for some forms of NiS could be 282^0 C.

Based on our factory inspection in the above study, there was no evidence to suggest that the heat soaking was not in accordance with the DIN --- in18516 standard and that the glass did not reach the hold temperature of $290^0 \pm 10^0$ C for 8 hours. Some researchers [3&4] have also reported that the rate of heating in the heat soaking process is also important, with slower rates being better than faster rates of heating.

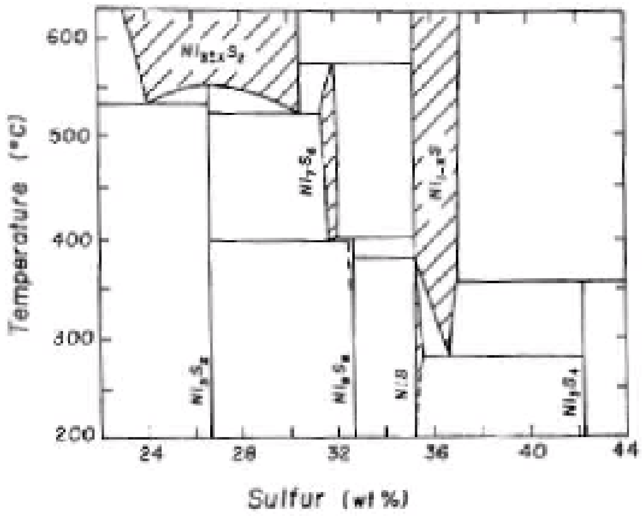
Based on the published literature [9] there is now ample evidence to suggest that there are many different forms of NiS, with differing rates of conversion from the α to the β phase. According to Kasper the presence of small amounts of iron in the NiS inclusion appear to slow down the conversion rate dramatically from the α to the β phase at the temperatures used for heat soaking. The transitional temperature for conversion from the α to the β phase is different for different forms of NiS according to Bishop [10].

It would therefore appear that the temperature of heat soaking should be maintained less than 282^0 C to ensure that conversion from the α to the β phase will occur more rapidly for all the possible forms of nickel sulphide inclusions. The chosen temperature of 282^0 C is based on the pure Nickel – Sulphur phase diagram on a theoretical basis and in manufacturing practice such a pure system may not be realistic. Furthermore, Sakai and Kikuta [4] concluded that the most suitable conditions for heat soak testing were:

1. Use a rate of heating less than 10 degrees Centigrade per minute.
2. Use a maximum temperature from 220 to 260 degrees Centigrade.
3. Keep the temperature at the maximum for less than 30 minutes.

Kasper [8] concluded that the heating rates should not be more than 2 degrees K per minute.

Fig 4



Conclusions

The glass industry needs to direct its resources to identify a suitable heat-soaking regime, which will have universal acceptance and provide the industry with a toughened glass totally free of NiS inclusions. Let us not eliminate the product because of a minor problem that occurs in batches of glass and manifests itself as a major problem on specific projects.

In real terms the percentage of NiS induced failures in comparison with the total population of all the toughened glass used in our buildings is relatively small. However, it is our responsibility to give the designer the confidence to enable the preparation of internationally acceptable specifications and standards. It is in our interest to ensure that we endeavour to minimise the potential risk of spontaneous fracture of both toughened and heat strengthened glass used in our buildings.

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