HEAT SOAKING AVOIDS SPONTANEOUS CRACKING OF THERMALLY TOUGHENED SAFETY GLASS

PD Dr. Andreas M. Kasper
SAINT-GOBAIN GLASS Deutschland, F&E Bauglas
52134 Herzogenrath, Germany

1. Keywords

1 = Spontaneous break, 2 = nickel sulfide, 3 = heat soak process, 4 = break probability

2. Abstract

Toughened glass applications on buildings occasionally suffer from so-called spontaneous fractures. If these are not caused by inadequate assembly or vandalism, in most cases a special kind of a glass inclusion, the so-called nickel sulfide stones, have been proved to be at the origin of the break.

Nickel sulfide (Millerite) is a mineral coming into the glass in very small quantity during the glass melting process. At temperatures below 350°C it undergoes a slow modification change, a so-called allotropic transformation, accompanied by a volume increase of 4%. Because of this specific mineralogical property it is able to cause sudden failure of toughened glass panes under certain conditions. These are: location of the inclusion in the tensile zone of the glass; minimum diameter of the inclusion (>50 µm); favourable temperature / time regime to allow the allotropic transformation to take place.

Since it is clear and proved by many laboratory results that the allotropic transformation of the dangerous inclusions is crucially influenced by temperature, a destructive process was developed called the Heat Soak Test. In every case it consists in heating up the panes to a certain temperature, holding this temperature for some hours, and cooling again to room temperature. Recently, in Europe, a new standard proposal (prEN 14179-1) was drafted. It fixes (290 ± 10)°C as the temperature the glass has to be kept on during at least two hours. The prEN also contains the exact prescription of the calibration procedure of the process oven to assure that at every possible load the glass in its entity is subject to the process conditions.

We did statistical calculations, based on more than 1200 breaks, observed in a pair of HST ovens working since a long time in accordance to the prEN, and on overall HST break statistics, taking into account more than 25000 tons of glass processed. This worst case estimation results that on a building with 10000 m² of heat soak processed glass the one year's break risk is less than 1%, i.e. in 100 years perhaps one glass may break because of NiS. How much less? We cannot tell it at the moment. Perhaps a lot...

3. Introduction

The spontaneous failure of thermally toughened safety glass is a long – known phenomenon which nevertheless is still very actual. Glass facades are beautiful, and the modern technologies to produce varying colours, reflections and tailored optical properties, and approved methods of fixing them on building facades, lead to a growing application of this aesthetically good-looking building component.

The reason for spontaneous breaks is well-known. Small inclusions of nickel sulfide being situated in the inner tensile zone of the tempered glass panes are subject to a slow allotropic transformation at room temperature which leads to a volume growth of these particles. Cracks form around them, and when reaching a certain critical length, they cause the sudden break of the entire pane. Up to now, the production of a float glass that would be absolutely free of nickel sulfide inclusions seems to be impossible.

To overcome the problem, the so-called heat soak test (HST) or Heat Soak Process was developed, a destroying process wherein the NiS inclusions shall be forced to transform at higher temperature of e.g. (290 ± 10)°C following the German DIN 18516[i] and the new draft European HST standard[ii], the latter being developed by CEN / TC 129 – "Glass in building" in 2001.

Although the HST is in use now since more than 30 years, it seems that its scientific background is not yet fully understood. In the last years several trials were undertaken to shorten the process, e.g. by raising the temperature level (so-called short HST) or by trying to stop the temperature during the cooling phase of the tempering process (so-called on-line HST), but these modifications were not successful and, when tried out in practise, caused epidemic failure of glass panes on the buildings concerned. The reason for these erroneous technical developments is a basic lack of knowledge about the real behaviour on heating or cooling of the NiS particulates surrounded by glass.

4. New DSC results

In our working group, we are treating the NiS and HST theme since 1994. We have published about 10 major articles since, and we found out real new things about it, e.g. that it is not a sulfur excess that causes the inclusions to act differently from chemically pure NiS, but iron traces (around 1% of the nickel) which are always present in the inclusions[iii], but they never contain a sulphur excess in reference to the total metal content[iv]. We measured the kinetic properties of these iron-containing nickel sulfides and found them to be very fast in comparison to the real HST breaks. The difference was explained by heating-up phenomena, but there is still a difference (of a factor of 3) when trying to fit the literature data published by MERKER[v].

With our modified method, we found slightly different kinetic parameters of the α to β transformation, the differences are significant and allowed us to fit very well MERKER's results.

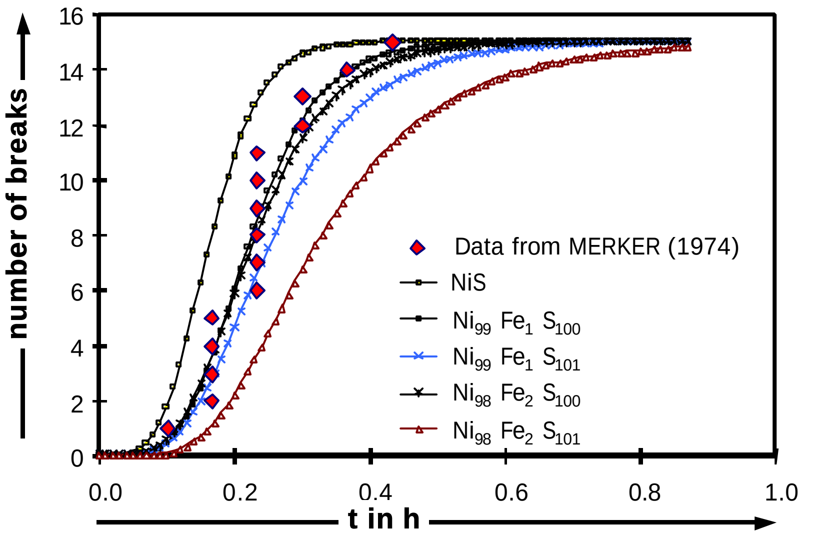

Figure 1: Our new kinetic results fit MERKER's laboratory results obtained from NiS inclusions he located in drawn glass sheets.

Nevertheless we are still unable to simulate the data of real HST ovens. There is another factor of about 3 left to be explained. We have come closer to the problem, but apparently it is not yet perfectly solved. Our actual research aims to find out the reason why; we think that in our laboratory trials we didn't respect all varieties of nickel sulfides really present in the glass[vi].

Until we will be able to really calculate break probabilities in HST ovens from the certified basic kinetic data, we must use the data we have obtained from the real HS process out of a very well known oven. This leads us to a worst case estimation of the break probability of HS processed glass.

5. Worst Case estimation of product safety after HST

The biggest part of the statistical data we are disposing of since 1996 are coming out of one oven type (i.e. four equally built HST ovens being operated at one site). In these, we were able to record the break times of 1258 panes. This high number seems to us to be sufficient to make confident statistical calculations. Figure 4 shows the overall WEIBULL[vii] evaluation of these 1258 breaks. It indicates the break probability. As an example, at time zero in the graph (when the holding temperature is reached), about 80% of the total breaks have already happened, and 20% will still happen. This means that every point on the curve indicates the remaining risk of a glass to break or not to break, depending on its residence time in the HST. It is clear that this is a statistical prediction to apply on a minimum number of glasses. For one single glass it has no useful meaning.

By extrapolation of the WEIBULL curve, one can estimate the statistical break probability after different holding times. In figure 2 it can also be seen that the heating rate of this oven type is low. It depends on the oven’s load and was meanly six hours (about one hour per ton loaded). Consequently, also under the light of the findings described in a previous communication[viii], we can be sure that in this oven the HST was done in an optimal way.

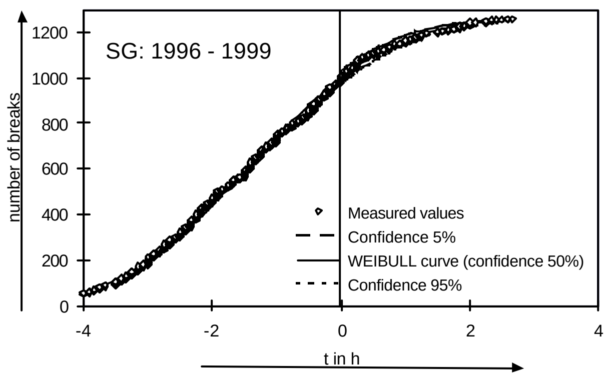

Figure 2: WEIBULL evaluation of 1258 breaks recorded in one type of HST furnace. Mean heating time was 6 h. When reaching constant temperature, 1012 breaks, i.e. 80.4% of the total number, have already happened.

Additionally, for a statistical calculation of the break probability of HS-Processed glass panes, the break probability of unprocessed float glass is needed. We actually have informations about the HST break rate of more than 25’000 tons of float glass from different production and tempering sites. The overall mean number is 8.7 tons per break (table 1), equivalent to 11.5 breaks per 100 tons of processed glass. We assume this figure to be representative also for real breaks on buildings, i.e. the HST to break exactly the glasses that would have broken spontaneously on buildings, although this seems to be a rather high estimation that is rarely observed on buildings equipped with unprocessed glass. To be really sure and conservative in our calculation, we assume the maximum number to be one break in six tons because some of the figures in table 1 are in this relatively high range.

Table 1: Break statistics in HST ovens.

	tons processed	breaks	t per break
A91-00	13569	874	15.5
B93-99	13642	2262	6.0
C	688	108	6.4
D	241	46	5.2
E	539	14	38.5
total	28679	3304	8.7

With the WEIBULL probability function and the break number in HST, it is now possible to calculate the break probability for a HS processed glass. For one single glass pane, this probability depends on its mass and on its residence time at the process temperature of 290°C. The longer it has been in the process, the lower its breaking probability is. On the other hand, the glass panes will not break all at the same time. A comprehensive estimation of the lifetime of glass panes would be 50 years. Also spontaneous breaks are reported to happen during the same time interval[ix]. This means that we may assume that the potential breaks happening with HS-Processed glass will spread continuously over the same time range.

Consequently, for the calculation of the one year’s breaking risk of one single glass pane, we derive the following equation:

$W_1 = m_1 * (W_{HST}) * [1 - \mathrm{WEIBULL}(t)] / L$

where	W_1	the single glass pane’s probability to break;
	m_1	the mass of this single pane;
	W_{HST}	the probability of breaking in the HST, given in breaks per ton;
	WEIBULL(t)	the 50% confidence level WEIBULL function of figure 4;
	L	Estimated lifetime of the glass pane.

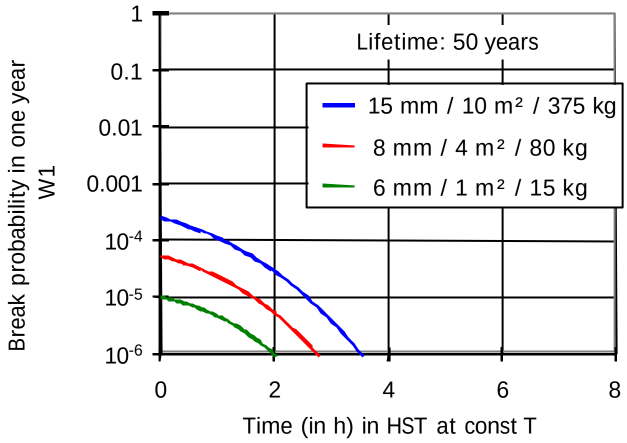

Figure 2: Calculated one year’s breaking probability for HS-Processed glass panes of different

The functions obtained, calculated for different glass pane's dimensions, are represented in figure 2. From those it becomes clear that the break probability decreases very fast with the residence time under process conditions, and after two hours at constant temperature of (290 ± 10)°C will be less than 1 break among 10000 panes of even big dimensions (375 kg).

Starting from this, calculations can be continued to estimate the break probability of the totality of "n" glass panes on a building. Following the laws of statistics the equation reads[1]:

$$W_n = 1 - (1 - W_1)^n$$

where W_n the probability to obtain a break on a given building;
W_1 the single glass pane's probability to break;
n the number of glass sheets on the building.

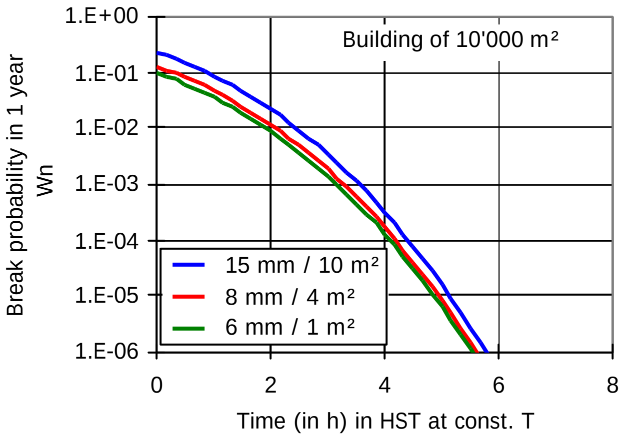

Figure 3: Calculated break probability for HS-processed glass panes on a building with 10000 m² of glass surface, as a function of the residence in HST time at a constant temperature of 290°C. After 2 hours of holding time, the probability is next to 0.01. This means that very probably no break will happen. The glass is safe.

1 Simplifying, to show the principle, we have supposed that all glass sheets of the building we are looking at are of the same shape. In reality this is normally not the case, but nevertheless the probability is calculated in the same way, grouping the glass sheets following their shapes, using the given formula, and summing up all obtained probabilities.

Figure 3 represents the functions obtained for a model building having 10000 m² glass panes of equal shape. The calculation was done for three cases of different thickness'. After little more than 2 hours of holding time, the break probabilities of even the model with the very thick glasses undergo 0.01. This means that then, the probability to have one break in a year is less than 1%. It can be said that under these conditions the glass is really safe.

This is why we clearly support the proposal of CEN-TC 129 – ”Glass in building” to limit the HST holding time to two hours, under the condition that also all of the other definitions regulated in the draft European standard are fully fulfilled. This means that the HST oven must be functionally tested by an independent institute with adequate glass loads; the glass temperature must be measured in a fixed number and position in the glass stack, and stated to have been between 280°C and 300°C during at least 2 hours on every point of the glass batch. Statistical quality control and repeated external certification must be applied. Then, the glass is as safe as calculated above.

6. Conclusion

Two hours of holding time in a heat soak process fulfilling the proposals of CEN-TC129 are enough for a sufficiently low break risk of the processed glass panes. Heat soaked thermally toughened safety glass, if processed following the European standard proposal prEN14179-1, really merits its name as a safe glass.

7. References

[i] German standard **DIN 18516-T4**: German Standard: Außenwandbekleidungen, hinterlüftet, Berlin: DIN (1990), p.2

[ii] European Draft Standard (**prEN 14179-1**): Heat soaked thermally toughened soda lime silicate safety glass - Part 1: Definition and description. CEN/TC 129, 11/2000

[iii] **KASPER, A., BORDEAUX, F.:** Nickel sulphide: New Results to Optimise the Heat Soak Test for Tempered Building Glasses. Glastech. Ber. Glass Sci. Technol. 73(2000)No.5 pp.130-142

[iv] **KASPER, A., STADELMANN, H.:** Zur Bildung von NiS in Glasschmelzen. Speach at the Fachausschuß III of DGG (German Glass Society), Oktober 2000, Würzburg; HVG-Mitteilung Nr.1981 (Dez.2000). Glastech. Ber. Glass Sci. Technol. 74(2001)no.3 pp. N35-36
KASPER, A., STADELMANN, H.: Chemical behavior of nickel sulfide in soda lime glass melts. Glastech. Ber. Glass Sci. Technol. 74(2001) (actually in press)

[v] **MERKER, L.:** Zum Verhalten des Nickelsulfids im Glas. (in German) Glastech. Ber. 47(1974)no.6, pp.116-121

[vi] **BARRY, J.C, FORD, S.:** An electron microscopic study of nickel sulfide inclusions in toughened glass. J. Mat. Sci. 36(2001) pp.3721-3730

[vii] German standard **DIN 55 303**, T.7 (Entwurf Juli 1993): Statistische Auswertung von Daten, Schätz- und Testverfahren bei zweiparametrischer Weibullverteilung. Berlin: DIN 1993

[viii] **KASPER, A.:** New measurements of NiS transformation kinetics to better understand the HST breakage data. Proceedings of the Glass Processing Days 2001, ISBN 952-91-3526-2.

Tampere, Finland, June 2001, pp.87-90. Conference speach, session 10.
http://www.glassfiles.com/library/lib_article.html?id=23

[ix] **FORD, T.:** Private communication (e-mail) concerning breaks on Australian buildings, January 2000