

Residual Risk of NiS Spontaneous Breakages after the Heat Soak Test

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Abstract

Spontaneous glass breakage of thermally toughened glass due to Nickel Sulphide (NiS) inclusions can be largely prevented by a preliminary Heat Soak Test (HST) (Kasper 2018a). This well-established test has been standardised in the EU Norm EN 14179-1:2005. However, according to literature (Kasper – Rubbert 2020), there is a small theoretical probability, that a potentially dangerous inclusion could lead to a breakage even after a well-conducted HST-test. In this paper, a statistical approach will be used to explain the level of residual risk of having a spontaneous breakage of heat soaked, thermally toughened, soda lime silicate safety glass due to the presence of critical nickel sulphide inclusions.

Keywords

Thermally toughened safety glass, Nickel Sulphide, Heat soak test, Residual risk

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1. Introduction

In terms of the chemical processes involved in glass production, the use of sulphate as the fining agent comes with a risk of it containing metal particles, so that nickel sulphide inclusion formation cannot be ruled out (Kasper et al. 2018c). Consequently, soda-lime silicate glass always contains nickel sulphide inclusions to a greater or lesser extent.

Due to the presence of nickel sulphide inclusions in the glass melt, spontaneous glass breakage of thermally toughened, soda-lime silicate safety glass is a well-known phenomenon. A recognised process to avoid spontaneous glass breakages on a building is the heat soak test (HST), which is described in the European standard EN14179-1. In this test, the thermally toughened glass is maintained for 2 hours at 290°C +/- 10°C or 260°C +/- 10°C, depending on whether the 2005 or 2016 version is used, prior to its installation. It is a test which should lead to the breakage of glass panes which have a critical nickel sulphide inclusion.

For several reasons, the heat soak test has not been universally adopted by glass processors. Some of them even advise against performing it, claiming it is theoretically not filtering 100% of all nickel sulphide inclusions (i.e. after the test there is a residual risk of having spontaneous breakages).

In the European standard EN14179-1 it is written that “the level of residual risk of spontaneous breakage of heat soaked thermally toughened soda lime silicate safety glass, on a statistical basis, due to the presence of critical nickel sulphide inclusions, is no more than 1 breakage per 400 tonnes of heat soaked thermally toughened soda lime silicate safety glass”. Too often, this sentence is wrongly interpreted as it is not asserting that 1 breakage per 400 tonnes is based on statistics.

Following a previous study which outlined a methodology to establish whether the heat soak test was done according to EN14179-1 (Letocart - Serruys 2025), determining the correct interpretation of the level of residual risk of spontaneous breakage of heat soaked, thermally toughened glass became a strong motivation for the present study.

2. Background

The generation of nickel sulphide (NiS) inclusions in the glass melt and its criticality for the stability of thermally toughened glass has been widely discussed in the literature (for example Kasper (2019)). In summary, the quenching process required to produce thermally toughened glass traps nickel sulphide inclusions into a high temperature hexagonal α -phase of NiS, which is not stable at room temperature. NiS is a reaction product of the glass melt with small metallic inclusions contained within the raw materials and cannot completely be avoided (Kasper et al. 2002).

Because this α -phase is metastable, it transforms slowly into a stable rhombohedral β -phase, which leads to a 2.5% increase of its volume at ambient temperature. If this inclusion is in the tensile stress zone within the glass thickness, the thermally toughened glass can break spontaneously, sometimes after years or decades.

To reduce the risk of spontaneous breakages on a building, the heat soak test (EN14179-1 standard), was introduced, and it is widely acknowledged that this test is effective in reducing the risk of a later spontaneous breakage to one breakage per 400 tonnes of heat soaked, thermally toughened, soda lime silicate safety glass. But what is the basis for this 1 breakage per 400 tonnes?

It is based on the Deutsche Bank Twin Towers in Frankfurt which was completed in 1984. The local building authorities specified that the maximum allowable level of spontaneous breakage, due to

nickel sulphide inclusions, must be less than 1 breakage in 400 tonnes of glass (i.e. 400 tonnes being the total quantity of thermally toughened glass in the façade). To fulfil this requirement, NSG Pilkington's German department developed the heat soak test. From the completion to the renovation of the Deutsche Bank Twin Towers in 2008, no breakages were recorded.

3. Interpretation of the breakage probability

Random events occur in clusters, so in any particular quantity of glass there may be more or less critical NiS inclusions than might be expected. To illustrate how randomness affects the numbers of incidents, the diagram in figure 1 shows 100 points randomly distributed (using a computer random number generator) in 100 squares, averaging 1 per square. There is one square with a cluster of 4 points and many squares have none. This is not an atypical event but has given rise to the idea of NiS inclusions occurring in batches (Colvin 2016).

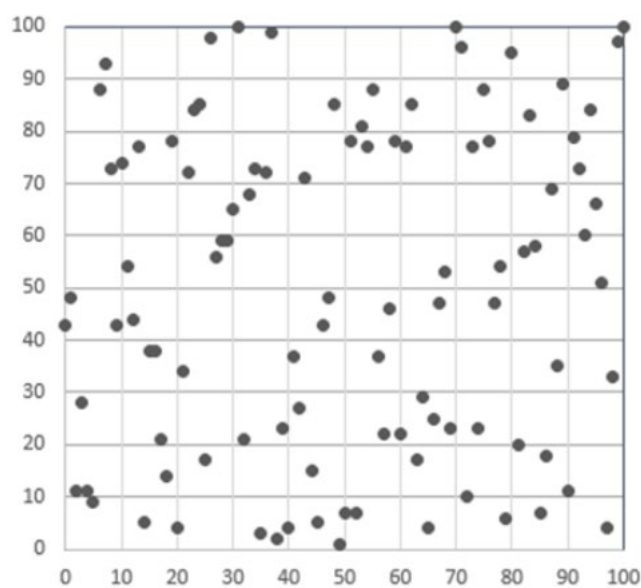


Fig. 1: 100 points randomly distributed (using a computer random number generator) in 100 squares, averaging 1 per square.

It is the view of the authors that the breakage probability should be interpreted in this way, i.e. it is a statistical approach considering an extremely large amount of data gathered over several decades. Therefore, it is not possible to claim that no breakages, or a maximum of one breakage, will occur because the quantity of glass used for the building is less than 400 tonnes of thermally toughened safety glass.

4. Determination of the potential number of breakages

Spontaneous breakages due to NiS inclusions happen at random and independently of each other meaning that the probability of one spontaneous breakage does not affect the probability of another spontaneous breakage. Moreover, the average rate of occurrence of spontaneous breakages does not change during the lifetime of the façade. These two factors are conditions enabling us to apply something known as Poisson distribution, which is a discrete probability distribution predicting the probability of the distribution of an event occurring a number of times in a given interval. In other

words, a Poisson distribution enables us to predict the probability of a spontaneous breakage, due to NiS inclusions, happening a number of times within a given volume of glass.

The probability mass function of the Poisson distribution is:

$$P = \frac{e^{-\lambda} \lambda^k}{k!} \tag{1}$$

Where:

- P is the probability that a spontaneous breakage will occur k times
- e is Euler’s constant (approximately 2.71828)
- λ is the average number of times a spontaneous breakage occurs (i.e. the residual risk on a statistical basis in relation to the total amount of glass)
- k is the number of times a spontaneous breakage occurs

4.1. Probability of having a spontaneous breakage where the residual risk is 1 per 400 tonnes

The reference residual risk of having a spontaneous breakage due to NiS inclusions in thermally toughened glass is given in the European standard EN14179-1 i.e. 1 breakage per 400 tonnes. This means that λ = 1 in the formula for the Poisson distribution in case the total amount of glass is 400 tonnes:

$$P = \frac{2,71828^{-1} \cdot 1^k}{k!} \tag{2}$$

Figure 2 shows the probability of having 0, 1, 2, etc. spontaneous breakages where the residual risk is defined being 1 breakage per 400 tonnes.

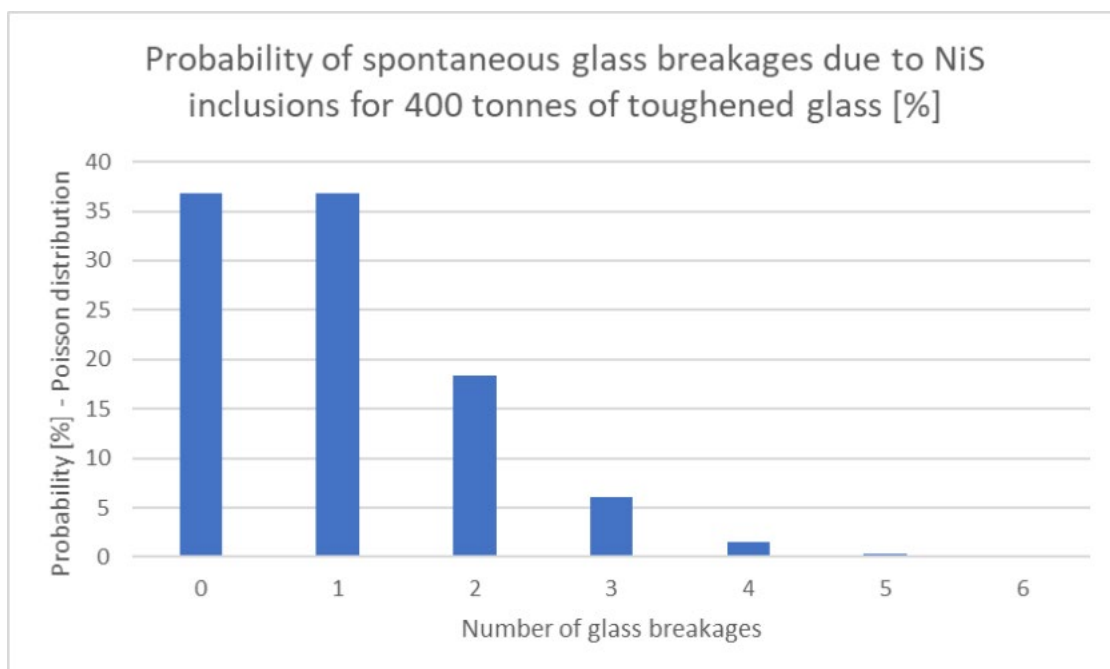


Fig. 2: Probability of spontaneous breakages due to NiS inclusions in thermally toughened glass. The horizontal axis is the index k, the number of spontaneous breakages. The vertical axis is the probability of k occurrences given λ = 1.

Often it is understood that, where the residual risk is 1 per 400 tonnes, there can only be 1 breakage where the total amount of thermally toughened glass is 400 tonnes. The calculation above shows that there is an equal chance of having 0 or 1 breakage but that there is also a chance of having 2 breakages (i.e. 18%) or even a very small chance of having 3 or 4 breakages (i.e. 6% respectively 2%).

Therefore, it is recommended that as soon as 1 spontaneous breakage occurs on a façade, it is documented and, if possible, the origin of the breakage, the so-called butterfly, is kept for further analysis if needed (fig. 3).

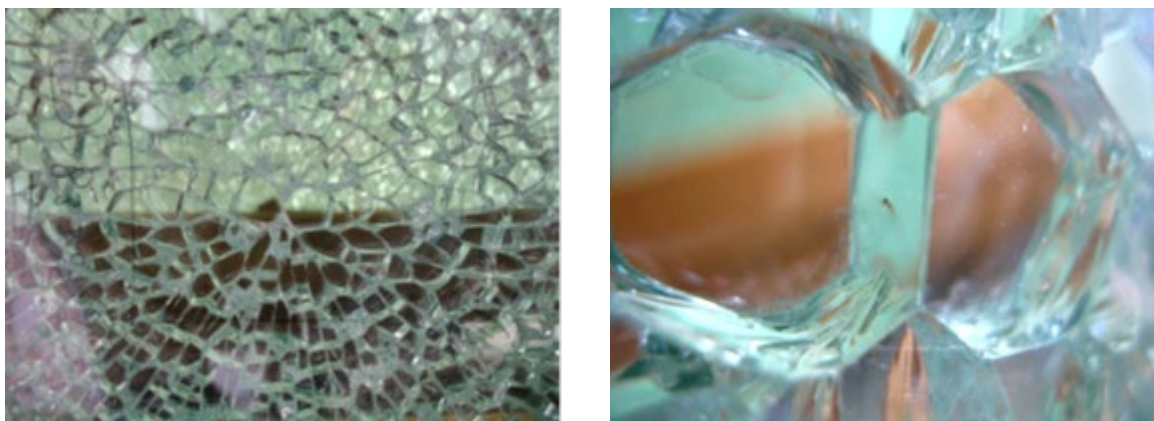


Fig. 3: Origin of a spontaneous breakage of thermally toughened soda-lime silicate safety glass. The typical “butterfly” with the inclusion in the middle.

4.2. Probability other than 1 per 400 tonnes

In a series of 4 publications in *Glass Structures & Engineering*, Andreas Kasper, PhD explains that during the heat soak test there are other impact factors, than the difference in thermal expansion (i.e. phase transformation) which may lead to breakage during the heat soak test (Kasper et al. 2018 / 2020). Two additional influencing factors may be the strong thermo-mechanical forces (temporary stress) induced into the glass during heating-up, and under-critical crack propagation driven by water diffusion at heat soak test temperature (Kasper et al. 2018a). Consequently, the heat soak test destroys more glass than would break on facades, as approximately 60% of the breakages observed in the heat soak test are not relevant for application on a building (Kasper et al. 2018a). The presumption that the breakage behaviour in the heat soak test and on buildings would be the same is clearly not correct (Kasper-Serruys 2002).

On this basis, he concludes that the statistical residual breakage risk of heat soak-tested, thermally toughened safety glass is estimated to be 1 in 6,000 tonnes of heat soak-tested glass (Kasper et al. 2018c).

Finally, a statistic evaluation of facade glass breakage records results in an estimated residual risk of only 1 breakage in 10,000 tonnes (Kasper 2018c).

Figure 4 shows the occurrence probability of a spontaneous breakage for the various residual risks (i.e. 1 per 400 tonnes; 1 per 6,000 tonnes and 1 per 10,000 tonnes). Based on the research of Andreas Kasper, it shows that heat soak tested, thermally toughened safety glass has a very low probability of having a spontaneous breakage due to a nickel sulphide inclusion.

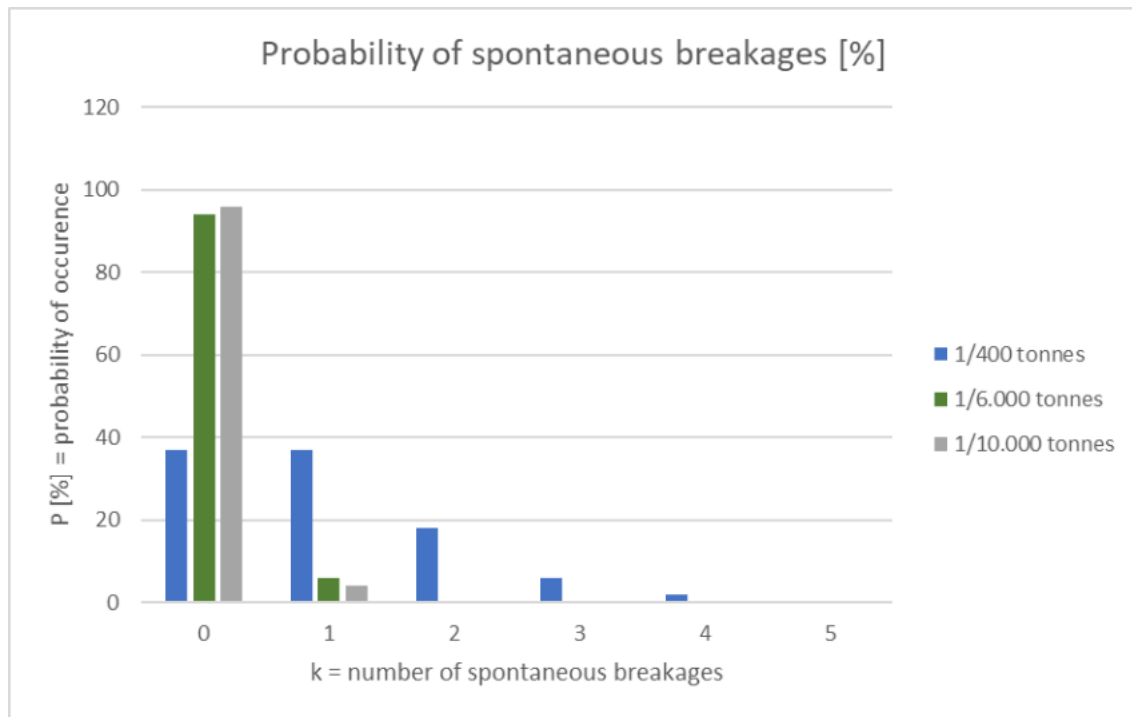


Fig. 4: Probability of spontaneous breakages due to NiS inclusions in 400 tonnes of thermally toughened safety glass. The horizontal axis is the index k, the number of spontaneous breakages. The vertical axis is the probability of k occurrences. The green dotted line is the reference according EN 14179-1. The green and grey bars are for estimated residual risks of 1 in 6000 tonnes respectively 1 in 10.000 tonnes.

4.3. Probability for un-heat soak-tested, thermally toughened safety glass

There is no doubt that average float glass quality is consistent, if batches from an exceptional contamination event are disregarded. Hence, an average conservative breakage probability of thermally toughened safety glass which has not been heat soak tested, is commonly said to be 1 breakage in approximately 6.0 tonnes (Kasper 2001). But this figure arose from the assumption that there was no difference between the breakages occurring during the heat soak test or on buildings. There were two reasons why this assumption was made. At the time, this difference seemed to be irrelevant and, secondly, the number of available butterflies from buildings was too low to make a statistically significant evaluation. As approximately 60% of the breakages observed in the heat soak test would not happen on site, the breakage probability of thermally toughened safety glass which has not been heat soak tested, is 1 breakage in approximately 10 tonnes.

Figure 5 shows the probability of having a spontaneous breakage due to NiS inclusions in 400 tonnes of thermally toughened safety glass which has not been heat soak tested. The residual risk being estimated at 1 breakage in approximately 10 tonnes of glass, means that, worst case, more than 50 breakages may happen.

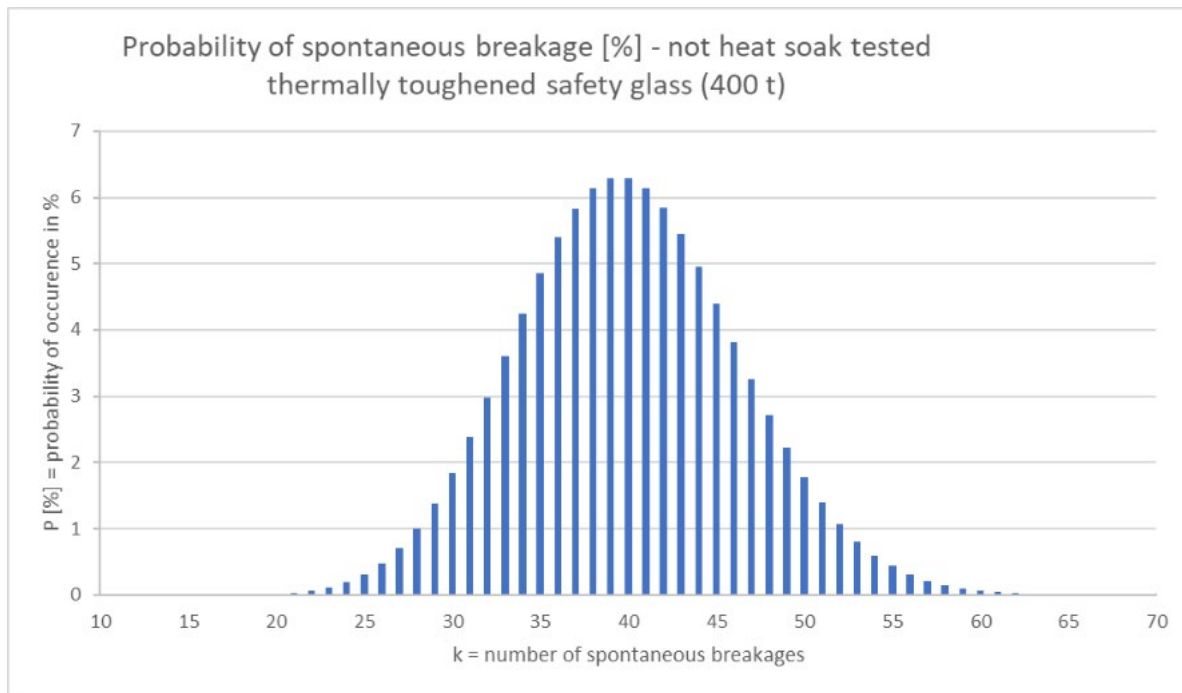


Fig. 5: Probability of spontaneous breakages due to NiS inclusions in 400 tonnes of thermally toughened safety glass which has not been heat soak tested.

4.4. Case study

A high-rise building has glass units comprising of 2 panes of 6 mm thermally toughened glass. There are in total 12,000 insulating glass units. The average dimensions of the insulating glass units are (1,4m x 2,6m) or approximately 3,6 m². The project is known by the authors. Since the renovation done in 2021, 130 spontaneous breakages were recorded.

Several butterflies were found and analysed. All the inclusions found were identified as being NiS.

The breakage rate is 1 breakage in 10.08 tonnes, which is nearly the same as would be expected for un-heat soak tested glass.

Figure 6 shows that the probability of the highest number of breakages has been reached, under the assumption that all breakages were due to NiS inclusions.

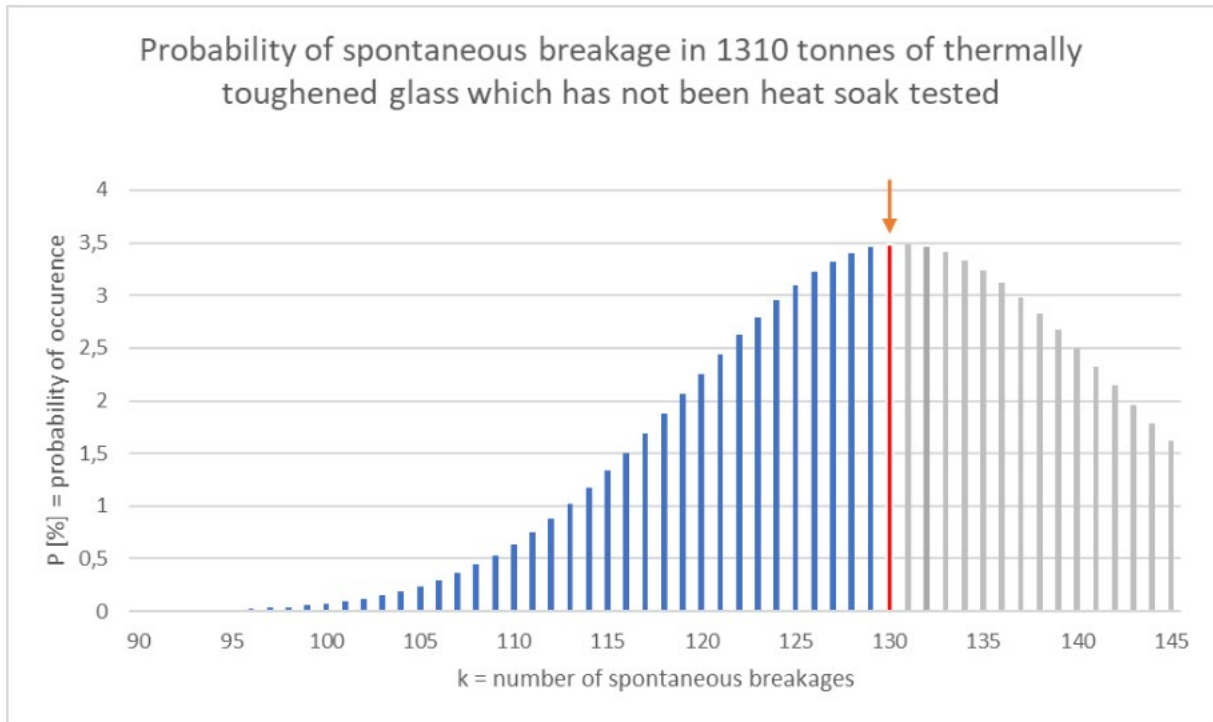
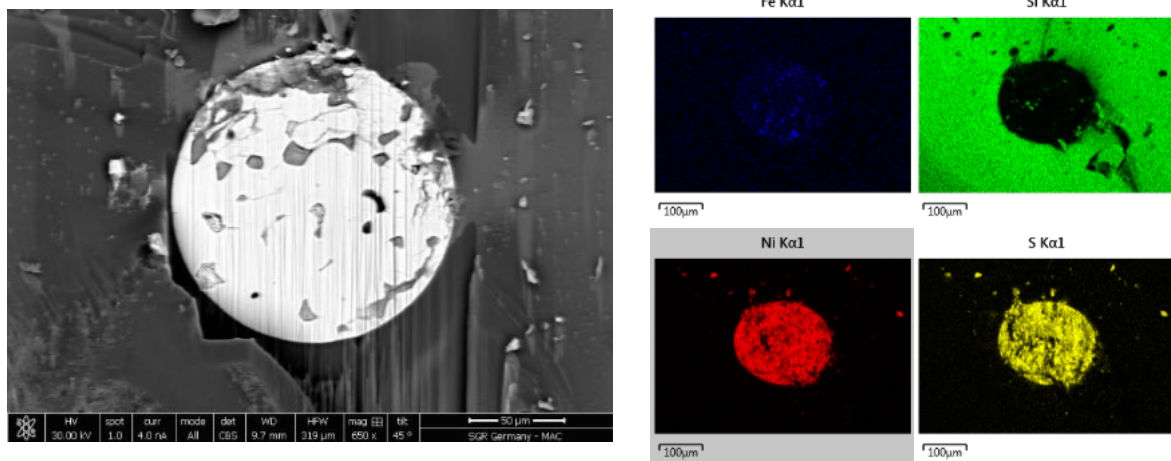


Fig. 6: Case study. 130 panes broken out of 12.000 insulating glass units. Under the assumption that all breakages are due to NiS inclusions, the residual risk is 1 breakage in 10 tonnes of glass and the highest probability has been reached.

Based on this probability study using the Poisson distribution, a first conclusion is that the heat soak test has probably not been done. The next step is to investigate whether the heat soak test has been done or not, by analysing the NiS inclusion(s) (Letocart – Serruys 2025). These analyses showed that the inclusions that caused spontaneous breakage do not belong to the categories of inclusions that have a theoretical chance of surviving the heat soak test and then causing spontaneous breakage on the facade. These categories of inclusions are: a higher amount of sulphur (over-stoichiometric case), a significant iron content, and inclusion compositions containing elements other than sulphur, nickel or iron). All inclusions were only made of the cold temperature, beta phase of millerite (nickel, sulphur and a small amount of iron). Based on this outcome it can be concluded that the glass supplied for this project was not heat soak tested.



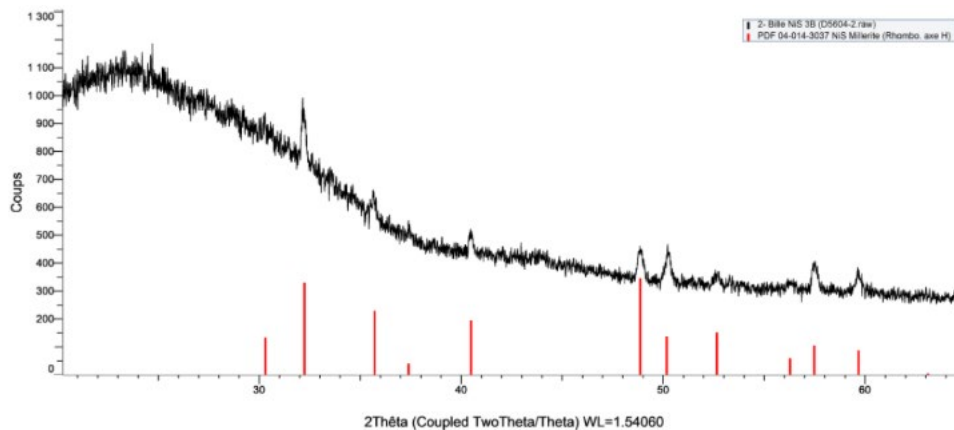


Fig. 7 : Detail of an inclusion after focused ion beam polishing showing the internal composition and morphology. Bottom image: corresponding micro-X-Rays diffractogram showing the presence of millerite only.

5. Conclusion

The sentence in the EN 14179-1 standard regarding the level of residual risk of spontaneous breakage of heat soaked, thermally toughened, soda lime silicate safety glass, on a statistical basis, due to the presence of critical nickel sulphide inclusions, is often misinterpreted. As spontaneous breakages, due to NiS inclusions, happen at random and independently from each other, the Poisson distribution can be used to determine the probability of having a spontaneous breakage in a defined amount of thermally toughened safety glass.

It is important, when thermally toughened safety glass is used on a facade, that from the very first spontaneous breakage, all is documented (e.g. date of breakage) and if possible, keep the breakage origin, the so-called butterfly. If more breakages occur, an approach using the Poisson distribution may enable us to draw a preliminary conclusion as to whether the heat soak test has been done. In a second step, breakage origins may be analysed in the laboratory (i.e. is the inclusion NiS? ; if yes, was the heat soak test done?) to conclude whether the heat soak test was done according to EN 14179-1 (Letocart – Serruys 2025).

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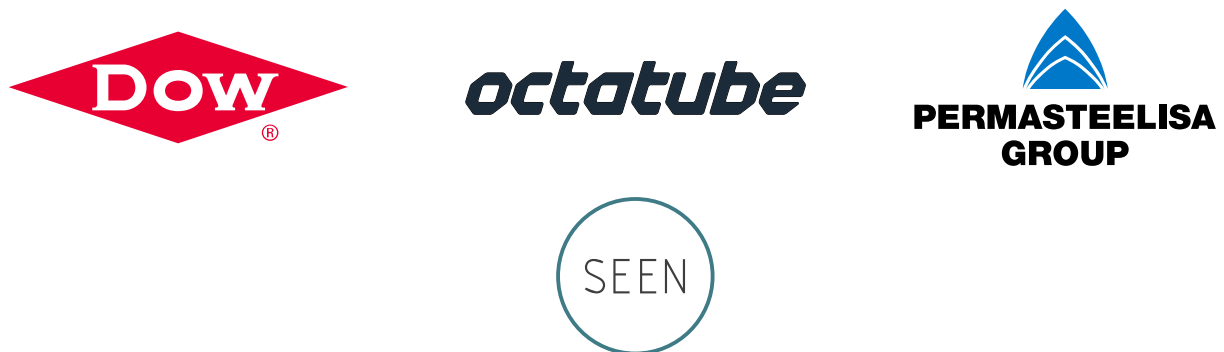
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