FACTORS THAT INFLUENCE SPONTANEOUS FAILURE IN THERMALLY TREATED GLASS – NICKEL SULPHIDE

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INTRODUCTION

Nickel sulfide (NiS) has been known to be the cause of spontaneous failure in heat-treated glass (Toughened/Tempered). The ICI House building in Melbourne, Australia, was one of the first buildings that used fully toughened (tempered) glass to overcome thermally induced edge cracking. An unanticipated consequence was that the glass cladding suffered NiS induced failures. The glazing was completed in September 1958. However, it was not until 1960 that failures where noted. Ballantyne[1] correctly identified the cause of the failures to be NiS inclusions.

Swain[2] on the basis of a simple fracture mechanics analysis identified a minimum critical size of inclusion below which failure could not occur.

Observations of numerous fractured heat-treated glass panels containing NiS has identified a new mechanism which explains the spontaneous failure. Measurements made on numerous NiS Stones have identified the importance of the mirror radius at the fracture origin and the residual tensile stresses in the heat-treated glass on the spontaneous failure mechanism.

The membrane stresses generated through the externally applied lateral loads from wind pressures can combine with the already identified stresses such as parabolic stresses introduced by the toughening process, stresses developed due to the phase change in NiS stones and stresses developed through environmental temperature.

Surface damage (scratches) to heat treated glass has also been identified as a significant source of failure. The failure characteristics from scratches can the similar to that generated from NiS stones. This has and can cause an erroneous conclusion as to the cause of the failures in heat-treated glasses.

THEORETICAL CONSIDERATIONS

The stresses that are developed or imposed around the NiS inclusion that causes spontaneous failure of the glass panels are potentially influenced by:

- the size of the inclusions,
- the purity of the inclusion,
- the location of the inclusion within the thickness of the glass,
- the toughening (tempering) stresses that encapsulated the inclusion,
- the environmental temperature to which the glass is exposed,
- the membrane stresses that are generated as a function of the wind loading, and
- the panel size and aspect ratio.

NiS is a crystalline material that is not miscible with molten soda line silicate glass. During the annealing process in the manufacture of float glass NiS is transformed from its high temperature α phase to the low temperature β phase. This transformation is accompanied by a 2% to 4% volumetric change in the stone and may or may not cause minute flaws to develop around the stone depending upon the temperature and the viscosity of the glass. In ordinary annealed glass, this is not a cause for concern. Generally, the NiS stone may become a problem, if situated very close to the glass surface and the glass panel is subjected to external tensile forces such as bending stresses in a window panel.

However, when annealed glass containing the NiS stone in the β phase is heated above the transition temperature (379^0 C) of the stone, it experiences a change from the low temperature β phase to the high temperature metabolic α phase. The fast cooling rates used in manufacture of toughened glass precludes a reversal of this phase change in the NiS stone. Consequently, the stone will naturally want to transform to the stable low temperature β phase. This is generally accompanied by the 2% to 4% volumetric increase

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discussed above. The previously identified vents can the further developed and propagated under the applied stresses at the glass-stone interface.

Swain[2] has shown that the stresses developed about a spectral isotropic inclusion are given by:

$$\sigma_r = 2\sigma_t = -P_o\,[R/r]^3\, r\, \mu\, R. \qquad (1)$$

Here P is the hydrostatic pressure and R is the radius for the inclusion, σ_r is the radial stress and σ_t is the circumferential or hoop stress.

The hydrostatic pressure can be determined from the following relationship:

$$P_o = -\{[(\rho_\alpha/\rho_\alpha)-1] / [(1+\nu_1/2E_1) + (1-2\nu_2/E_2)]\} \qquad (2)$$

Here, E_1 and E_2 are the Poisson's ratio and the Young's modulus of the host material and the inclusion respectively.

PROPERTIES OF NiS AND GLASS

Property	Glass	NiS	
		α	β
Young's Modulus E (GNm^{-2})	73	80	70
Poisson's Ratio	0.22	0.27	0.2
Density (Kg m^{-3})	2.49×10^3	5.46×10^3	5.25×10^{-3}
Coefficient of Expansion $^0C^{-1}$	77.5×10^{-7}	163.0×10^{-7}	145×10^{-7}

Table 1– Mechanical &Thermal Properties of NiS & Glass

Using the values mechanical and total properties of NiS as shown in Table 1, the hydrostatic pressure resulting from α to β phase transformation of NiS in Soda Lime Silicate glass can be calculated to be 858 MPa. This pressure is in the immediate vicinity of the glass-NiS interface. Its value drops away from this interface. This pre supposes that the NiS stone is completely transformed from its α phase to β phase and too many impurities that can reduce the magnitude or the stresses generated.

DIFFERENTIAL THERMAL EXPANSION

The difference in the rate of thermal expansion between NiS and the glass substrate will also reduce stresses within the inclusion. The stresses that result due to differential thermal expansion coefficient is given by:

$$\begin{aligned}\sigma_r = 2\,\sigma_t = &\{(\alpha_{glass} - \alpha_{NiS})\,(T_g - T) / [\{(1+\nu_1)/w\,E_1\} + \\ &\{(1-2\,\nu_2 / E_2)\}X(R/r)^3 \qquad (3)\\ = &\;P_\alpha\ (R/r)^3\end{aligned}$$

Using the material properties listed in Table 1, the value for P_{ot} is -231.8 MPa for a mean value of α_α and α_β for NiS

Hence, the stress at the surface of the stone is given by:

$$\begin{aligned}\sigma &= (858 - 231.8)\,(R/r)^3\\ &= 627\,(R/r)^3\ \text{MPa.}\end{aligned}$$

TEMPERING STRESSES

The process of heat treating glass includes a parabolic stress distribution in the glass plate. The maximum tensile stress in the middle is half the induced surface compressive stress. International design codes require a minimum level of 68.9 MPa surface compression. This in turn means that there is a minimum of 34.47 MPa tension in the body of glass. It is in this tensile stress region that the NiS inclusion becomes a problem. The stress across the thickness of the glass is not uniform. Consequently the position of the stone relative to the thickness of glass will determine the influence that tempering stresses has on the spontaneous failure of the glass. For instance, it is unlikely for any NiS inclusion located in the compressive zone of the heat-treated glass to fail spontaneously as a result of the phase change transformation that occurs in the NiS inclusion.

Normally commercial grade heat-treated glass has a surface compression of approximately 100 MPa. This in turn means that there is 50 MPa internal tension. The philosophy of using heat-strengthened glass (ie, glass with a lower level of internal tension) has been found to minimise the risk of spontaneous failure.

MEMBRANE STRESS

Generally when glass is used in buildings as windows it is subject to lateral pressures from wind. When the deflection of the glass panel exceeds 75% of its thickness, the stresses induced in the panel gradually changes from bending to membrane, ie, in-plane stresses. The aspect ratio of the plate is critical in determining of the in-plane stresses. Numerous researchers, including Vallabhan et al[3] have theoretically computed the membrane stresses in glass plates for various aspect ratios. The in- plane stresses acting in the vicinity of the stone can enhance the nucleation of the original flaw that was probably developed by the expansion of the NiS in its transition from α to β phases.

The rate of this nucleation will be a function of the location of the NiS inclusion in the body of the glass and its position relative to the wind induced membrane stresses developed under load. When the stone is closer to either of the surfaces, then the application of bending stresses will contribute to the growth of the crack at that position more than when it is at the centre. On the other hand, irrespective of the location, the membrane stress is a direct additive to the maximum tension that is in existence in the body of the heat-treated glass panel.

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With regard to the position of the stone relative to the total panel, it is well understood that the stress distribution from laterally applied load moves from the centre of the panel towards the corners as the load is increased creating membrane stresses. So the influence of the membrane stress at the crack tip will be a function of the magnitude of the lateral load, size of the panel and the position of the stone in the panel relative to the centre.

FRACTURE MECHANICS CRITERIA

An analysis of the fracture surfaces in the vicinity of the stone indicates the correlation between the mirror radius and the magnitude of the fracture stress. Liu[5] and Bansal[6] have identified a correlation of flaw size to stress at fracture in glass.

$$\sigma_f = K_{ic} / a^{1/2} \qquad (4)$$

a = Stone diameter

The use of these techniques in analysing the failure with NiS is not relevant, as the correlation expected does not occur. (Ie, there is no relationship between the size of the stone (flaw) and the magnitude of the stress at which failure occurred.)

On the other hand, if the mirror radius were related to the fracture stress by the following equation, then good correlation is obtained.

$$\sigma_f = K_{ic} / C^{1/2}$$

K = Critical stress intensity factor=0.584 MNn (Jacob[4])

C = Mirror radius from fracture surface

Further analysis using fracture mechanics criteria and internal stresses in fully toughened glass indicates that there is a direct relationship between inclusion size and internal stresses.

SURFACE DEGRADATION (SCRATCHES)

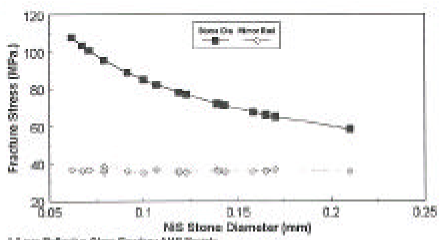
The influence of surface scratching on the strength of heat strengthened glass is an area requiring further investigation to fully understand the fracture mechanics involved.

Window glass by its intrinsic nature must be periodically cleaned during the life of the building. The process of cleaning the window glass is likely to create minute surface scratches on the glass. The extent of these scratches is dependent on the condition of the glass surface prior to the cleaning operation and the severity of the cleaning process.

Generally most glass manufacturers specify an acceptable limit to the number and extent of surface scratches in the glass supplied. This is supported by various specifications defining acceptable scratch limits in new glass.

INVESTIGATION OF FAILURES

A total of 40 NiS samples courtesy of the AMP Society (Australia), were carefully examined to determine the NiS stone diameter and the associated mirror radii. All the samples came from the same building and in all probability was toughened to approximately the same level. The result of measurement of stone diameter and mirror radi, and the calculated fracture stresses using the stone diameter and the mirror radii are summarised in Table 2. Figure 1 is a plot of these calculated fracture stresses (estimated from equations 4 and 5) and the stone diameter. The noteworthy feature is the plot between the fracture calculated from the mirror dimension is independent of the stone diameter. This suggests that the influence of all the associated stresses generated by the phase change acting together around the stone to cause the spontaneous failure rather than the stresses generated by the phase change only. This clearly shows that the fracture stress is a function of the mirror radii and not the stone diameter. It also indicates that the flaw associated with the NiS will only become critical after it has grown to a certain amount beyond which spontaneous fracture will occur irrespective of stone size.



* 3 mm Reflective Glass Courtesy AMP Society

ANALYSIS OF NiS SAMPLES

N°	Date Of Failure	NiS Dia	Mirror Radius	Fracture Stress	
				Stone Dia	Mirror Rad
1	1/91	0.17	4.3	65.27	38.93
2	2/91	0.14	5.5	71.92	34.42
3	12/91	0.21	4.5	58.72	38.96
4	15/91	0.1	5.25	85.1	35.23
5	18/91	0.08	4.8	85.1	36.85
6	19/91	0.08	5.25	95.74	35.23
7	20/91	0.01	4.8	77.68	36.85
8	22/91	0.17	4.7	66.25	37.24
9	28/91	0.08	4.8	95.74	36.85
10	4/92	0.92	5	88.96	36.1
11	10/92	0.14	4.3	72.18	38.93
12	13/92	0.17	5	66.25	36.1
13	28/92	0.11	4.8	82.27	36.85
14	2/93	0.14	437	71.92	37.24
15	3/93	0.12	5.2	76.73	35.4
16	4/93	0.07	4.2	103.5	39.39
17	7/93	0.12	3.5	78.01	36.1
18	8/93	0.07	4.5	106.9	38.06
19	19/93	0.16	4.6	67.4	37.64
20	20/93	0.14	5.5	71.16	34.42

Table 2 NiS sample giving stone diameter and the fracture stress calculated from the mirror radius and stone diameter.

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Table 2 relates to a building (courtesy AMP Society) where failures have been observed over a three-year period. The only consistency in the data is the mirror radius and hence the fracture stresses computed using the mirror radii.

Swain[2] in his paper considered a simple parabolic stress distribution in the body of the toughened glass. His analysis did not include any externally applied stresses. He derived a relationship between the size of the NiS inclusion and internal stress in toughened glass.

Using the data from Table 1, the critical size of flaws can be computed as:

0	70MPa	60MPa	50MPa	40MPa	30MPa	20MPa
R	22	27	36.5	51	78	114

Here, σ_0 is the tension in the fully toughened glass.

Normally commercially supplied fully toughened glass has a surface compression of approximately 100 MPa, which relates to 50 MPa internal tension. Consequently, for normal tinted or reflective toughened glass installed in a building and subject to wind loading, the total stress acting around the stone could be 60 MPa or more. This comprised of 50 MPa internal tension, 10 MPa of membrane stress, 2 –3 MPa of temperature stress, and phase transformation stresses.

Various researches' references and glass companies have generally observed that the critical NiS flaw sizes is 110 microns below which spontaneous fracture should not occur. In theory however, smaller stones can cause spontaneous failure if the stress field is enhanced by external forces such as membrane stresses, temperature stresses. For example, a panel 3.3m x 1.3m if subjected to a wind pressure of 2.45 kPa, the theoretical membrane stress that will develop in the panel will be about 13 MPa. With the addition of internal tension (50 MPa) a stress field of 63 MPa around the stone will develop. This indicates that smaller diameter stones could fracture spontaneously.

A building in Sydney has suffered NiS failure for the past 30 years. The significant point of this project is that the panel sizes are relatively small and the membrane stresses are low and are obviously not contributing to the crack growth associated with the NiS stone. Theoretical computations indicated that the maximum membrane stress generated in these panels is 5.34 MPa. This is a relatively low value in comparison with the tempering stresses encapsulated within the body of the glass, consequently not contributing to the growth of the existing vents associated with the NiS stone.

MEMBRANE STRESSES

DWP r/kPa	Panel Size/m x m	Glass Thickness/mm	Membrane Stress MPa
2.5	2.5 x 2.0	6	20.8
2.5	2.0 x 2.0	6	8.26
2.5	2.0 x 1.5	6	10.5
2.5	1.5 x 1.0	6	5.34
2.5	2.5 x 1.5	6	13.38
2.5	2.5 x 1.0	6	5.92

Table 3: Membrane Stresses in monolithic glass plates of different sizes.

Table 3 provides the theoretical maximum membrane stresses in monolithic glass plates of different sizes subject to a maximum design wind pressure of 2.5 kPa. It can be seen that the membrane stress build up is a function of wind pressure, glass thickness and aspect ratio of the panel.

CONCLUSION

The general classification of toughened glass requires a minimum residual surface compression of 68 MPa, which equates to an internal tension of about 34 MPa. Environmental temperature hence glass temperature, glass type, glass thickness, panel size, and aspect ratio influence the process of manufacturing toughened glass. Consequently, most manufacturers tend to over temper the glass to ensure compliance with specifications. Unfortunately, the code provides an upper limit for the surface compression and hence tensions in the body of the glass. The general industry consensus is to toughen glass to a surface compression of approximately 100 MPa that provides for an equivalent tensile stress in the centre of the glass of approximately 50 MPa. This is significant and is a pre-condition that facilitates the spontaneous failure due to NiS which has been subjected to 3 different forces:

- Volumetric expansion of the stone due to phase change;
- Relative thermal stresses due to the different coefficient of expansion between the stone and the glass;
- The external induced membrane stresses due to lateral pressures applied to the window panel.

It is concluded that the NiS stones, which have been blamed for majority of toughened glass failures, is not the only cause. The externally induced membrane stress through lateral pressure is also a significant contributor to crack growth and subsequent spontaneous failure.

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The fact that spontaneous failure occurs on window panels with no relevance to environmental conditions such as wind pressures and high temperature supports the significant role played by the membrane stress. Membrane stresses can cause slow crack growth around the stone. When this crack reaches a critical level, spontaneous fracture will occur. This critical limit is approximately the mirror radius. This in turn is directly related to the size of the crack initiated in the vicinity of the stone and to the magnitude of the tensile stress in the glass as a result of the toughening process and other applied or developed stresses. Figure 1 is a cumulative graph of all the stones used in this study. The scatter of the fracture stress using the mirror radius clearly indicates the influence of environmental conditions that is, membrane stress on the failure mechanism.

For the typical example considered the membrane stress of 13 MPa developed due to wind load must be added to the magnitude of the tensile stress that exists in the toughened panel. This is now the combined tensile force that is acting around the NiS inclusion. The degree of phase change that has occurred will also influence the stress field. This in turn reduces the critical stone (NiS) diameter that can spontaneously fracture.

Considering Sample N^o 1/91 (Table3) the stress at the point of bifurcation (crack branching) is determined as 0.0387 MPa using equation 3. This is extremely low and can be considered negligible. Consequently other stresses must be available for further crack propagation. These are:

- The tensile stress in the toughened glass, and
- The membrane stresses.

It has generally been observed that the critical NiS flaw size is 100 microns below which spontaneous failure should not occur. This study shows that smaller stones can also be responsible for spontaneous failure if external forces like membrane stresses enhance the stress field.

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