Thermally toughened safety glass: correlation between flexural strength, fragmentation and surface compressive stress

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Keywords

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Abstract

The aim of the present research is to define the correlation between the following parameters in heat treated glass:

1. flexural strength tested according EN 1288-3;
2. fragmentation tested according EN 12150;
3. surface compressive stress tested according ASTM 1279

The research is the development of a previous one carried out at Stazione Sperimentale Vetro (SSV) [1, 2], which was focused mainly on the correlation between surface compressive stress and flexural strength. In the present paper those experimental data are increased and the correlation is extended to fragmentation for un-coated and coated glass. The correlation between surface compressive stress and mechanical strength or fragmentation could be relevant for the producer, who may also use surface pre-stress measurement as a means of a non destructive product control. The relationship between above mentioned parameters is evinced and may be used as reference for the production conformity in Factory Production Control, to confirm or not as it's now stated in the relevant EN 's which require to compare time by time the measured value versus actual test results.

Considering the sampling dimensions used for the present work, however these correlations have to be validated also for normal production dimensions, especially in case of heat strengthened glass fragmentation evaluation. The influence of rollers on glass surface damage has to be taken in account because this aspect could affect the mechanical strength despite appropriate surface compressive stress level.

Introduction

SSV tested thousands of glass panes from 2000 according the European Standards for thermally toughened safety glass EN 12150 and according EN 1883 for heat strengthened glass.

Figure 1 Fragmentation pattern in thermally toughened safety glass (a) and in heat strengthened glass (b).

From the 1st September 2005 due to the implementation of the harmonized part of the standards, ITT certification became compulsory according CPD 89/106 requirements and the amount of tests increase.

The required procedure to evaluate the flexural strength is described in EN 1288-3:2000 (four point bending strength test), whereas the surface pre-stress is carried out by SSV laboratory following the ASTM C1279:2000 (and the following revised versions). The fragmentation test is carried out with the same procedure in thermally toughened safety glass and in heat strengthened one, but the fragmentation requirements differ due to the different pattern, as evinced in figure 1. The correlation between surface compressive stress and fragmentation should be carried on only for thermally toughened safety glass, while between surface compressive stress and flexural strength should be carried on for both glass typologies. The ITT requirements and surface stress measurements are evaluated on specimens with dimensions 360 mm x 1100 mm. The specification of tests procedure and instruments and testing machine used to carry on the tests are reported in the previous paper [1].

Some authors tried to define a theoretical correlation between surface compressive stress and fragmentation[3, 4, 5], and others tried to found it by test on thermally toughened safety glass [6] or heat strengthened glass [7], or both [2, 8]. These correlations could be useful for producers of thermally treated glass to show compliance with requirements of Standards as they give the opportunity to use a non destructive method, like the measurement of surface compressive stress, to verify the performance of produced glass. It can be also an advantage for the final client which could verify the heat treatment quality.

Up to day, only the ASTM C1048:2004, "Standard Specification for Heat-Treated Flat Glass - Kind HS, Kind FT Coated and Uncoated Glass", specified a surface compressive stress requirement as showed in table 1, where are reported also the typical value for annealed glass [9, 10], and the bending strength limits defined in EN Standards.

The process of thermal treatment of float glass panes induces a surface pre-compression of glass for a thickness of 0.21 t, where t is the pane thickness, and the stress profile is parabolic with the following expression:

Glass type	Surface Compression (MPa)	Bending Strength (MPa)
Annealed	< 4	>45
Heat strengthened	24÷52	>70
Thermally toughened safety	>69	>120

Table 1 Thresholds for surface compressive stress, following ASTM Standards, and bending strength, following EN standards.

$$\sigma_x(z) = \sigma_y(z) = \sigma_c \cdot \left(1\text{-}12 \cdot (z/t)^2\right) \quad (1)$$

where σ_c is central tension at mid-plane; z is the axis perpendicular to pane plane, defined by the x and y axes. For the value z=t/2, corresponding to pane surface, it is evinced that the surface compressive stress is equal to:

$$\sigma_x(z) = \sigma_y(z) = -2 \cdot \sigma_c \quad (2)$$

Gulati in [4, 5] formulated a fragility model, considering a square pattern of fragmentation and the stored elastic energy during tempering process. It is evinced that the stored elastic energy is more than double when the central tension increases from to (0.7 σ_0) to σ_0, where σ_0 is the strength of annealed glass, prior to tempering. High central tensile stress increases the glass strength but increases also its fragility. In [5] the term of "fragmentation density" was introduced as the number N of particles per unit area of pane and it was evinced that N depends on the 4th power of central tension, as it is independent of plate thickness. These aspect were considered in the analysis of SSV data collected during the wide time period of testing.

Data base for correlations

Data was mostly collected from 2002 up to 2010 and when possible were organised firstly on base of the test type in:
- surface compressive stress ($\sigma_{s(ASTM\text{-}1279)}$): evaluated by GASP laser on five points on tin side following EN 12150-2:2004.
- fragmentation test ($n_{(EN\text{-}12150)}$): number of particles evaluated on a square area 50 mm x 50 mm in the coarsest zone
- 4 point bending test ($\sigma_{b(EN\text{-}1288)}$): considering both air/tin side, or coated/uncoated side, in tension.

Data are collected by year and then arranged by thickness; do to the fact that not always surface compression measurements is required and performed, only samplings with the all tests were considered for the correlation, so that each point represents a single specimen. The total amount of specimens analysed are reported in table 2 and 3.

Thickness	$\sigma_{s(ASTM\text{-}1279)}$ related to $\sigma_{b(EN\text{-}1288)}$
4 mm	52
5 mm	59
6 mm	237
8 mm	212
10 mm	219
12 mm	92
15 mm	52
All thickness	923

Table 3 Specimens tested for each glass pane thickness for correlation $\sigma_{s(ASTM\text{-}1279)}$ versus $\sigma_{b(EN\text{-}1288)}$

Data are in mainly representative of thermally treated glass production in Italy, with same sampling from others European producers. During these last years specimens with soft coated glass increase due to the rapid evolution of energetic requirements of glass for building applications (low emissivity, selective products, ect.), and new temperable products appears on the market, for this reason all type of glass are here considered.

As data refers to different producers it means the tempering process differs for ovens and their technology of heating and convention if one, from tempering recipes related to glass thickness and type; sometime also weather conditions (barometric pressures and daily temperature) can influence the level of tempering.

Correlation between surface compressive stress and fragmentation for thermally safety glass

The all data of specimens with surface compressive stress and particles number were divided in groups on the base of fragmentation density, defined as:

$$N = {n_{(EN-12150)}} \Big/ {A_{(EN-12150)}} \quad (3)$$

where $A_{(EN\text{-}12150)}$ is the area of the square 50 mm x 50 mm in which the particles are counted.

The minimum particles amount for thermally toughened safety glass is reported in EN 12150:2000. The data were collected by:
- N: <0.7(10); <0.8(20); <1.6(40); <2.4(60); <3.2(80), <4.0 (100); 4.8(120); <5.6(140); <6.4 (160); <7.2(180), in the round brackets the corresponding number of particles are reported;
- $n_{(EN\text{-}12150)}$: for each group the mean value of particles was evaluated
- $\sigma_{s(ASTM\text{-}1279)}$: for each group the mean value of surface compressive stress was evaluated.

This procedure was applied for each glass thickness and in figure 2 it is plotted the mean value of each group $\sigma_{s(ASTM\text{-}1279)}$) versus $n_{(EN\text{-}12150)}$.
The correlation between the plotted quantities is defined by a linear regression, which equation is defined as:

$$y(x) = m \cdot x + q \quad (4)$$

This is an approach to modelling the relationship between a scalar variable Y, in this case $n_{(EN\text{-}12150)}$, and the variable denoted X, $\sigma_{s(ASTM\text{-}1279)}$. In linear regression, data are modelled using linear functions, and unknown model parameters are estimated from the data. The linear regression model is fitted using the least squares approach. In table 4, the parameters of linear regression and the coefficient of determination (R^2) value of the experimental data are reported.

Not conform specimens were also considered in the proposed correlation, because the aim was to define the lower limit of surface compressive stress for appropriate fragmentation of safety glass pane. Limit number of particles is 40 for the tested panes, except 15 mm glass thickness for which the limit is 30. The correlation line for 4 mm, 5 mm, 6 mm, 8 mm and 10 mm glass pane are quite similar, whereas a lower slope coefficient is evinced for 12 mm and 15 mm glass pane. This may be due to the increment of thickness that may asks more elastic energy to induce cracks, because of the compressive layer is bigger than in thinner pane, as well from a "cluster" effect which does not allow a finest fragmentation. From figure 2 it is evinced that -generally- the surface compressive stress should be more than 85÷90 MPa to define the thermally toughened safety glass as safety glass. This value varies not much considering different thickness;

Thickness	$\sigma_{s(ASTM\text{-}1279)}$ related to $n_{(EN\text{-}12150)}$	$n_{(EN\text{-}12150)}$ (conform)	$n_{(EN\text{-}12150)}$ (not conform)
4 mm	280	259	21
5 mm	180	171	9
6 mm	378	365	13
8 mm	345	317	28
10 mm	330	282	48
12 mm	170	151	19
15 mm	69	59	10
All thickness	1752	1604	148

Table 2 Specimens tested for each glass pane thickness for correlation $\sigma_{s(ASTM\text{-}1279)}$ versus $n_{(EN\text{-}1288)}$

then the authors propose a safety limit value of 90 MPa, independently from glass thickness. It is evident that the increment of fragmentation density for glass panes thicker than 10 mm needs more stored elastic energy. This could be reached increasing the surface compressive stress, but at the same time the glass pane becomes more brittle. This aspect has to be taken into account during the design of glass structural elements.

There are cases with a quite high measured surface compressive stress with an amount of fragments lower than the expected one, sometime just in the limit or immediately below. This phenomenon is quite rare and related to coated glass. We interpret it as due to a strong non homogenous velocity of cooling between the two surfaces and from these and the glass core; more investigation needs to found a method to detect these anomalies.

Despite it is not possible to find a similar correlation for heat strengthened glass, experience shows that it conforms to EN 1863-1:2000 fragmentation requirements when surface compressive stress is between 35 MPa and 55 MPa.

Correlation between surface compressive stress and flexural strength

In such correlation the two kind of thermally treated glass was considered.

Data of specimens with surface compressive stress and flexural strength measurement were firstly divided by glass thickness; then the specimens with only tin side and un-coated side in tension in the bending test were selected, because this is the side where $\sigma_{s(ASTM-1279)}$ was measured. Moreover the data were segregated considering specimens with only central fracture origin. The plotted value carried out from experimental data are:

- surface compressive stress considered as mean value of five measure for each specimen;
- flexural strength calculated at collapse load, following the equation defined in EN 1288-3:2000.

Few values of specimens of not-heat treated tested as received from the float line process, were also added.

The diagram of figure 3 shows clearly the type of glass that were tested: annealed, heat strengthened and thermally toughened safety glass. The relationship between the scalar variable $\sigma_{b(EN-1288)}$, and the one denoted $\sigma_{s(ASTM-1279)}$ is still a linear regression, which equation is plotted in figure 3. The limit value of surface compressive stress that has to be reached to respect the characteristic strength value of thermally toughened safety glass is 80 MPa; in the case of heat strengthened glass, this

Thickness	Slope coefficient (m)	Intercept coefficient (q)	R^2
4 mm	4,38	- 343,95	0,99
5 mm	6,00	- 509,81	0,99
6 mm	4,49	- 344,74	0,76
8 mm	3,15	- 239,59	0,97
10 mm	4,28	- 348,60	0,82
12 mm	2,05	- 146,75	0,98
15 mm	1,56	- 101,95	0,91

Table 4 Parameters of linear regression in the correlation $\sigma_{s(ASTM-1279)}$ versus $n_{(EN-12150)}$, and coefficient of determination R^2 for experimental data.

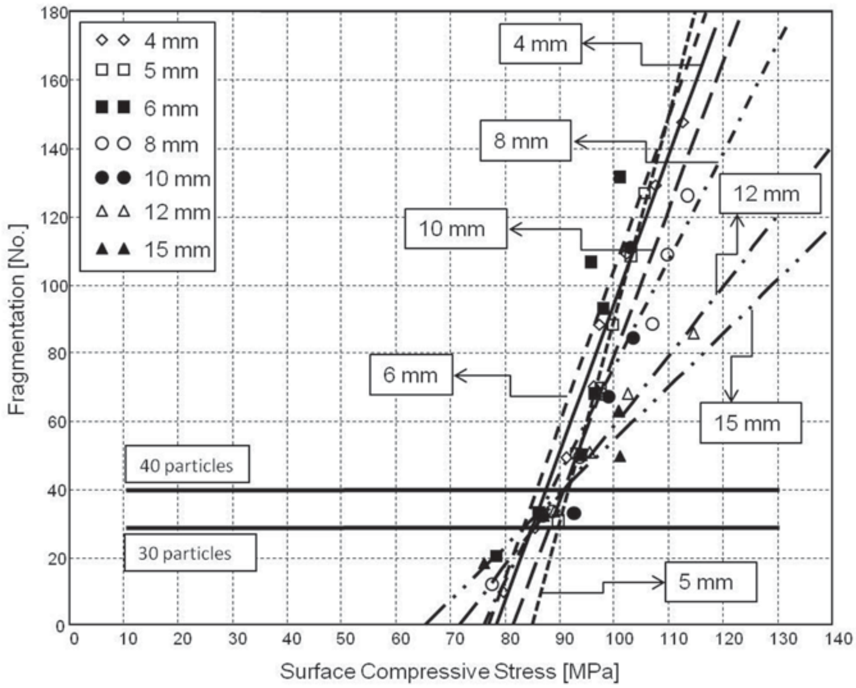

Figure 2 Correlation of surface compressive stress versus fragmentation for each glass thickness, considering thermally toughened safety glass, coated and un-coated, as conform and not conform specimens.

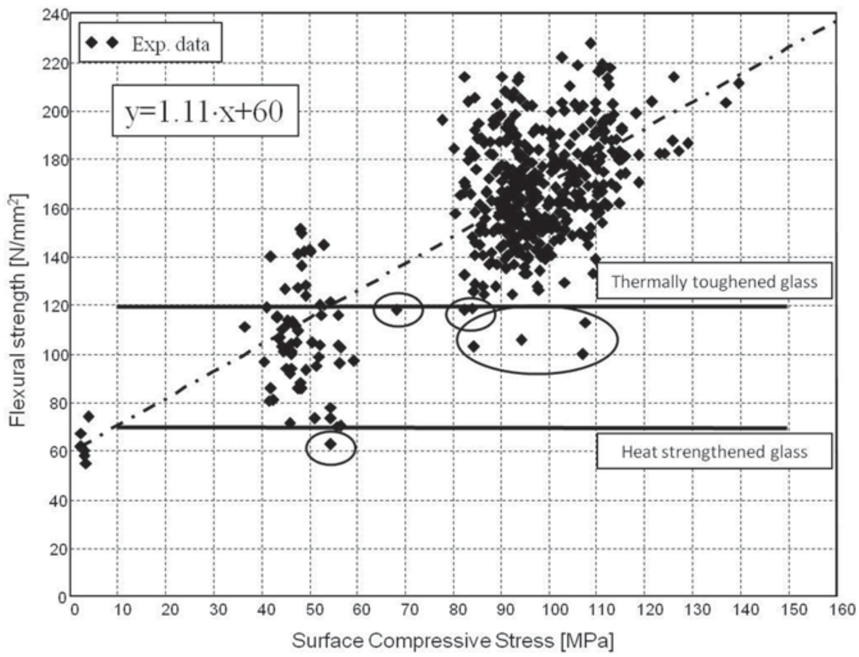

Figure 3 Correlation of surface compressive stress versus flexural strength for the all tested glass thickness, considering tin side and un-coated side only, conform and not conform specimens. The circled data were not considered for the correlation, because it was evident by fracture analysis that the tensile side was damaged. The coefficient of determination R^2 for experimental data is equal to 0,58.

value is 35 MPa. Also considering the all data collected of mechanical strength (air side and coated side in tension), these values can be confirmed. These value confirm also the previous research [2], increasing the number of tested specimens.

Conclusions

The analysed data covered about many years of laboratory tests on different type of glass (annealed, heat strengthened and thermally toughened safety) provided by different producers in Italy and in Europe.

Correlations was carried out considering the possibility to identify a non destructive procedure to evaluate the conformity of glass products to European Standards. This procedure was defined as the measurements of surface compressive stress on tin side, as prescribed by EN 12150-2:2004 for thermally toughened safety glass. Correlation between this parameter to fragmentation density and to flexural strength was evaluated by the application of linear regression method. The not conform specimens were considered too, because they could occur in production and must be detected in the procedure.

1) Surface compressive stress and fragmentation density correlation is generally independent from glass thickness, except for pane thicker than 10 mm. The elaboration of experimental data evinced that the minimum level of surface compressive stress that needs to be induced by tempering process is:

- 90 MPa in glass pane thickness less or equal to 10 mm;
- 85 MPa for thickness bigger or equal to 12 mm;

to get good fragmentation and define the glass product as "safe".

Gulati in [5] find experimentally a correlation between fragmentation density and the 4th power of tensile mid-plane stress. This correlation was verified according the data collected by the authors. However, the analysis of experimental data shows that may be expressed also by linear equations only for thermally toughened safety glass, and reported in the following:

$$n_{(EN-12150)}\left(\sigma_{s(ASTM-1279)}\right)=4.5\cdot\sigma_{s(ASTM-1279)}-357$$

for 4 mm ≤ t ≤ 10 mm (5)

$$n_{(EN-12150)}\left(\sigma_{s(ASTM-1279)}\right)=1.8\cdot\sigma_{s(ASTM-1279)}-121$$

for 12 mm ≤ t ≤ 15 m (6)

2) Surface compressive stress and flexural strength correlation was carried on considering all sampling thickness tested with tin side and not coated side in tension; these sides are the ones where the surface compressive stress was measured by GASP laser. The results carried out are in agreement with those obtained in the previous investigation [2] and the general equation is:

$$\sigma_{(EN-1288)}\left(\sigma_{s(ASTM-1279)}\right)=1.11\cdot\sigma_{s(ASTM-1279)}+60 \quad (7)$$

The limit value for surface compressive stress to obtain conform specimens in terms of flexural strength is independent by pane thickness; the more reliable mean value is 90 MPa for thermally toughened safety glass and 45 MPa for heat strengthened one. These are safe values because they give corresponding flexural strength higher than that defined as characteristic value by the Standards of references.
Additional tests should be done on annealed and heat strengthened glass to increase the population of samples for these type of products.

Another aspect, that has to be considered, is the fragmentation of heat strengthened glass for which a different parameter needs to be assumed (es. particle length or weight), to complete the correlation in terms of surface compressive stress and fragmentation.

The results obtained till now needs to be verified on current production glass pane dimensions to confirm the validity independently from pane size.

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