Thermally Processed Glass: Correlation Between Surface Compression, Mechanical and Fragmentation Test

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Keywords

Surface Compression, Bending Strength, Fragmentation, Thermally Treated

Abstract

The paper correlates the data recorded in Stazione Sperimentale del Vetro, in many years. The aim is to define a correlation between the following parameters in heat treated glass:
1. bending strength tested according EN 1288-3 [1];
2. fragmentation tested according relevant Standards [2, 3, 4];
3. surface compression stress measured with laser Gasp [5]
For heat strengthened glass the fragmentation correlation due to the different crack path ("island" fragments instead of small fragments) is only related to conformity: C/NC.
The research is the development of the previous ones [6, 7] carried out at Stazione Sperimentale Vetro, increasing the experimental data (up to 2016) considering in detail the emissivity of coated glass and extending also to enamelled glass.
The correlation between surface compression stress and mechanical strength and fragmentation is relevant for the manufacturer, who may use surface pre-stress measurement as a means of product control.

Introduction

Thermally treated glass is used in many applications and the range of glass products is quite wide considering coated glass and enamelled too.
Coated glass needs to respect energetic parameters: low-e, selective and reflective glass in function of the climatic zone and law requirements for specific projects.
In the recent years the enamelled glass has started to be request more and more for specific applications in which the designer would like to hide some elements or create an opaque surface or for artistic propose.
The enamelled treatment could be applied uniformly on the whole surface or at specific zones (i.e. along the glass pane edges), according to drawings (screen printing) or pattern (points, lines, strips). They are produced by applying and burning a coloured paint on glass surface; then the pane is thermally treated. The interaction between glass surface and paint is a tricky aspect due to the tensile stress that the frit induces at the interface and by the effect of pigment granules [8]; both weaken the surface of application. This aspect is taken in account by Standards reducing the minimum values for the mechanical strength [2, 3, 4]. In Italy a new Standard was published at the beginning of 2017 [9].
SSV carries out many experimental tests on these products. The data are collected to evaluate a correlation between the Surface Compression Stress (SC) and the other characteristics: Fragmentation (FR) and Flexural Bending Strength (FB). This database started in 2002 and it is still going on. The data reported in the present paper had been collected until the end of 2016 and had been organised as:
1. surface compression stress tested according [5]
2. bending strength tested according [1];
3. fragmentation tested according relevant standard [2, 3, 4];
The aim of this paper is to evaluate and extend the considerations carried out in the previous papers [6, 7, 10] to coated and enamelled glass. Furthermore heat strengthened glass data were considered, whereas fragmentation is considered in terms of conformity Y/N according [2] due to the different crack path ("island" fragments instead of small fragments).
The correlation between surface compression stress and mechanical strength and fragmentation is relevant for the manufacturer, who may use surface pre-stress measurement as a means of product control.

Thermal process on heat treated glass

The soda lime silicate glass HS (conformity to [2]) or TT (conformity to [3, 4]) is a glass in which was induced permanent surface compressive stress through a controlled process of heating and cooling to increase mechanical and thermal strength; for TT product, in addition, to get the fragmentation characteristics such as to limit the damage to people and/or things in case of its failure.
The heat transfer in the tempering process takes place through:
- Radiation (resistors in the pre-heating and heating)
- Conduction (contact with the rollers)
- Convection (important in the case of coated glass)

The convention plays a crucial role in the process with introduction of the low-e glass in the market: glass with high emissivity absorbs heat while one with low emissivity reflects it. The presence of a face with lower emissivity may involve an asymmetrical heating and the resulting curvature of the pane at the end of the treatment, with unlikely no homogeneous residual stresses.
After heating, in the first instants of air blowing, the glass surface is cooled more quickly than the centre of glass pane and, in few seconds due to the low thermal conductivity, the temperature difference between the surface and the core of the pane reaches the maximum value. It is evinced that more energy is requested to temper thin glass than that for thicker one. The quenching step is obtained by forced blowing whose time depends on the glass thickness.
Undesired residual stress on glass surface may be caused mainly by:
- no uniformity of heating of pane in its plane and between the two surfaces
- different quenching speed from point to point of pane
- presence of holes, notches, that induce differential heating and quenching rate

It is necessary to control the process at every stage to avoid these problems.

Measurement of residual stress in heat treated glass

The measure of residual stress has to be carried on by photoelastic measurement, which has been widely developed in the recent years. Nowadays, the main instruments are: 1) GASP, registered trademark of Strainoptics Technologies; 2) SCALP, developed by GlasStress Ltd.
The measurement is carried on to evaluate the SC and correlate this non-destructive measure with the FB of FR values carried out by destructive tests. Redner wrote many papers on this topic [11, 12, 13, 14, 15, 16]

explaining the features of the GASP instrument and its capability to be used in QC after glass tempering. Other authors proposed a new instrument (SCALP) based on scattered light polariscope technique [17, 18] evincing that the residual stress in tempered glass can be highly inhomogeneous, both locally and globally.

Frame of the research

The research is developed according to test procedure reported in:
- EN 12150-1 [3] for thermally toughened glass, in the following named TT
- EN 14179-1 [4] for heat soaked thermally toughened glass, included in TT
- EN 1863-1 [2] for heat strengthened glass, in the following named HS

which prescribe fragmentation test (FR) and four point bending test (FB), according [1], after measurement of surface compression stress (SC), according to [5].
The value carried out from experimental data are:
- SC: surface compressive stress considered as mean value of five measure for each specimen;
- FR: number of fragments obtained according to [3, 4]
- FB: flexural strength calculated at collapse load, following the equation defined in [1].

The SC is correlated to FR and FB respectively.
Up to day, the ASTM C1048:2012 [19] and ISO Standards [20, 21] specify a surface compressive stress requirement as showed in table 1; whereas the EN Standards define the bending strength limits and the minimum number of fragments as reported in table 2.
The assessment for FR differs between HS and TT glass because the crack path is different. Therefore in case of HS glass the only indication of Conformity (C) or not (NC) has been considered to evaluate the minimum SC necessary to get it. In case of TT glass the number of particles have been considered according the count procedure of Annex C [3].
All the specimens were grouped as reported in tables 3 and 4, where the number of available tested specimens are reported for the two correlations.
EN Standards define B1 as coated glass with 0.89 ≥ ε > 0.25. In this range a wide set of products exists and the heat treatment differs greatly from glass to glass. For this reason the authors divided in B1 (ε = 0.89) and B1_bis (0.89 > ε > 0.25), but also B1_bis ε range is too large.
Data are representative of thermally treated glass production in Italy, with some sampling from other European producers.
As data refers to different producers, it means the tempering process differs for ovens and their technology of heating and convention, as for tempering recipes related to glass thickness and type.

Standard Reference	Heat Strengthened	Thermally Toughened
EN 1863-1:2012	No value is indicated	--
EN 12150-1	--	No value is indicated
EN 14179-1:2016	--	No value is indicated
ASTM C1048:2012	24÷52 MPa (thickness equal or lower than 6 mm)	69 MPa
ISO/DIS 22509 rev.:2016	25÷55 MPa	--
ISO/FDIS 12540:2016	--	80 MPa minimum for FB 90 MPa minimum for FR

Table 1 Reference Value of Surface Compressive Stress

Standard Reference	Float and coated	Enamelled
EN 1863-1:2012	70 N/mm² (FB)	45 N/mm² (FB)
EN 12150-1:2015	120 N/mm² (FB)	75 N/mm² (FB)
EN 14179-1:2016	120 N/mm² (FB)	75 N/mm² (FB)
Glass thickness 4÷12 mm 5 mm	40 TT (FR) 30 TT (FR)	40 TT (FR) 30 TT (FR)

Table 2 Minimum value of Bending Strength and number of fragments for TT

Thickness (mm)-**HS**	4		5		6		8		10		12		15		Total	
	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC
Float	35	5	68	3	134	6	129	21	106	26	45	10	--	--	517	71
B1: ε=0.89	--	--	5	0	10	15	5	5	5	0	5	0	--	--	30	20
B1_bis: 0.25<ε<0.89	--	--	--	--	5	0	5	0	5	5	5	0	--	--	20	5
B2: 0.1<ε<=0.25	--	--	--	--	20	0	5	0	--	--	0	5	--	--	25	5
B3: ε<=0.1	--	--	15	0	25	5	15	5	23	10	0	5	--	--	78	25
Enamelled	--	--	10	0	8	0	3	0	5	10	--	--	--	--	26	10
Thickness (mm)-**TT**	4		5		6		8		10		12		15		Total	
	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC
Float	248	27	252	19	283	15	286	19	310	55	227	38	104	10	1710	183
B1: ε=0.89	30	0	20	0	95	0	65	0	30	0	--	--	--	--	240	0
B1_bis: 0.25<ε<0.89	20	0	5	0	53	7	56	9	60	5	--	-	--	--	194	21
B2: 0.1<ε<=0.25	37	3	10	0	55	0	30	0	15	0	10	0	--	--	157	3
B3: ε<=0.1	99	12	25	0	92	13	136	14	85	10	13	2	--	--	450	51
Enamelled	30	0	14	0	15	0	7	3	25	0	5	0	--	--	96	3

Table 3. Number of specimens for SC vs FR

Thickness (mm)-**HS**	4		5		6		8		10		12		15		Total	
	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC
Float	19	0	57	0	102	0	87	0	83	0	31	2	--	--	379	2
B1: ε=0.89	--	--	3	0	8	0	5	0	3	0	3	0	--	--	21	0
B1_bis: 0.25<ε<0.89	--	--	--	--	4	0	4	0	4	0	4	0	--	--	16	0
B2: 0.1<ε<=0.25	--	--	13	0	24	0	--	--	--	--	--	--	--	--	37	0
B3: ε<=0.1	--	--	11	9	40	0	53	0	31	-	2	0	--	--	137	9
Enamelled	--	--	--	--	--	--	--	--	17	0	--	--	--	--	17	0
Thickness (mm)-**TT**	4		5		6		8		10		12		15		Total	
	C	NC	C	NC	C	NC	C	C	C	NC	C	NC	C	NC	C	NC
Float	119	0	140	1	162	1	146	2	221	1	145	2	82	0	1015	7
B1: ε=0.89	21	3	15	0	82	0	66	0	20	0	--	--	--	--	204	3
B1_bis: 0.25<ε<0.89	15	0	4	0	33	1	40	0	52	0	--	--	--	--	144	1
B2: 0.1<ε<=0.25	25	0	7	0	51	0	20	1	11	0	8	0	--	--	122	1
B3: ε<=0.1	81	0	25	0	90	1	126	4	95	1	23	5	--	--	440	11
Enamelled	70	5	30	0	24	0	4	0	50	4	7	0	--	--	188	9

Note: The FB specimens are lesser because, if the sampling did not pass FR, the test was stopped. For this reason the NC specimens are also limited.

Table 4. Number of specimens for SC vs FB

			SC (MPa)		FB (N/mm²)	
Producer	Glass Type	Tensile side	Mean	Dev. St.	Mean	Dev. St.
A	10 mm Clear Float TT	no roller	107.0	6.8	194.4	23.8
		roller	106.0	7.4	138.2	8.3
B	10 mm Clear Float TT	no roller	105.5	1.9	202.0	26.8
		roller	104.6	2.2	165.0	18.1
	10 mm Clear Float HS	no roller	43.7	2.4	129.8	11.9
		roller	43.8	0.9	81.7	10.1

Table 5. Data of float glass

Another aspect concerns the rollers influence on glass bending strength. It is well known the influence of "tin" and "air" side referred to float glass due to the rollers effect during the annealing phase. Sometime this effect is also amplified when the "tin" side is placed in contact with tempering rollers and the process is not well controlled. The authors carried out specific tests on some producer plans to evaluated the roller effect both for float (Tab. 5) and enamelled glass panes concerning the bending strength.

In these two very extremely cases tempering roller effect is clearly evident. The SC values are equal inside the same sampling but the bending strength differs between "roller" and "no roller" side, independently from the "air" or "tin" side. In general the decrement of bending strength is coupled by a decrement of standard deviation: defects, introduced by the roller, reduce data dispersion. The correlations of this paper (see tables 9-11) will be also affected by this effect.

The enamelling process weakens the glass surface and this aspect is well known, whereby the Standards define lower value of characteristic bending strength for enamelled glass, as reported in table 2. Usually the paint is applied on the "air" side and then the glass is processed bonding the paint to the glass surface. In this way the "tin" side is in contact with rollers. The enamelling process reduces the bending strength and the value dispersion too.

Fragmentation vs Surface Compression

All the data of specimens (from 4 mm to 15 mm glass thickness) with recorded surface compressive stress and particles number were considered and the minimum acceptable value of SC to get the conformity was recorded and reported in table 6 in function of glass thickness and type for TT but with a certain degree of NC incidence. The data are plotted in figure 1 (float glass), 2a, b, c, d (coated glass), and 3 (enamelled glass).

The authors proposed in the previous paper a safety limit value of 90 MPa, independently from glass thickness, considering only float glass. This value should be confirmed by the increment of test data for float, B1 and B2. It may be revised considering the coated b1_bis, B3 and enamelled glass, which request higher SC to reach conformity; for these the value should be increased to 95 MPa (Tab. 7). Also at this limit values some specimens have high SC but they are not conform (see % incidence), especially for B3, where 100 MPa will reduce the NC incidence. The reason could be that the SC is measured at tin side and the SC should be not homogeneous along the glass thickness, giving NC fragmentation pattern.

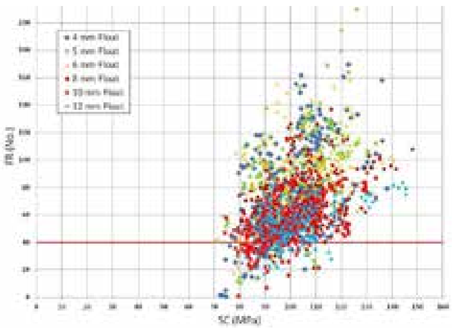

Figure 1. Correlation of surface compressive stress (SC) versus fragmentation (FR) for float glass

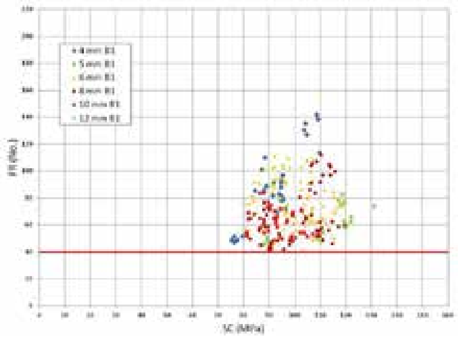

Figure 2a. Correlation of surface compressive stress (SC) versus fragmentation (FR) for B1 coated glass

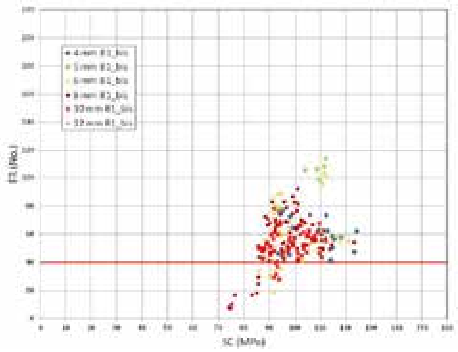

Figure 2b. Correlation of surface compressive stress (SC) versus fragmentation (FR) for B1_bis coated glass

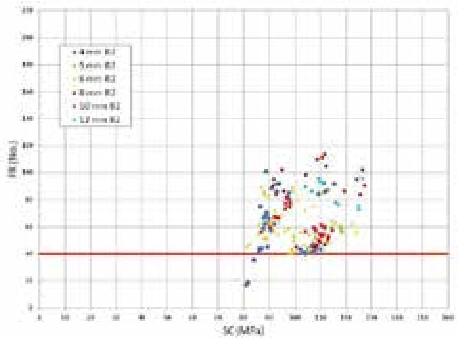

Figure 2c. Correlation of surface compressive stress (SC) versus fragmentation (FR) for B2 coated glass

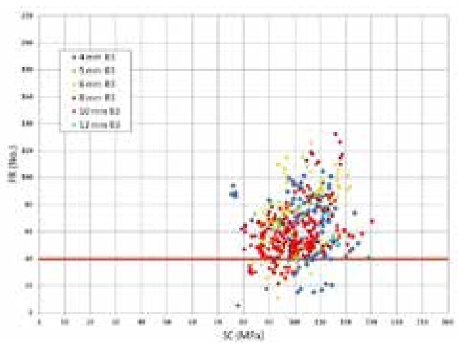

Figure 2d. Correlation of surface compressive stress (SC) versus fragmentation (FR) for B3 coated glass

	4 mm	5 mm	6 mm	8 mm	10 mm	12 mm	15 mm
Float	80(7%)	80(6%)	80(4%)	80(6%)	80(14%)	80(14%)	80 (1%)
B1: ε=0.89	75	88	81	83	81	--	--
B1_bis: 0.25<ε<0.89	94	--	87(10%)	86	86(8%)	--	--
B2: 0.1<ε<=0.25	86	--	81	87	92	104	--
B3: ε<=0.1	99(11%)	85	86(11%)	79(9%)	86(5%)	108	--
Enamelled	96	97	97	91	96	--	--

Note: (%) incidence value of data in the limit SCvalue but NC to FR.

Table 6. Minimum value SC (MPa) vs conform FR for TT in SSV specimens

	Limit value SC	4 mm	5 mm	6 mm	8 mm	10 mm	12 mm	15 mm
Float	90	2	3	2	3	6	11	0
B1: ε=0.89	90	0	0	0	0	0	--	--
B1_bis: 0.25<ε<0.89	95	0	--	0	0	0	--	--
B2: 0.1<ε<=0.25	90	0	--	0	0	0	0	--
B3: ε<=0.1	95	11	0	6	1	4	13	--
Enamelled	95	0	0	0	0	0	--	--

Table 7. Incidence value (%) of NC data for SC (MPa) vs FR in TT with the proposed SC value

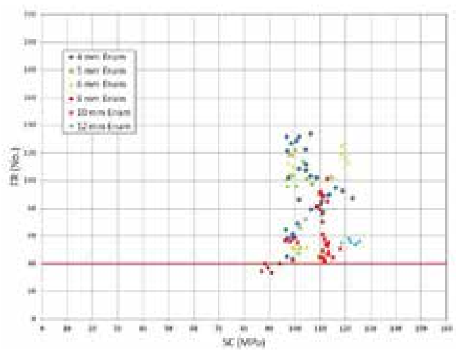

Figure 3. Correlation of surface compressive stress (SC) versus fragmentation (FR) for enamelled glass

	4 mm	5 mm	6 mm	8 mm	10 mm	12 mm	15 mm
Float	67	65	62	63	58	60	--
B1: ε=0.89	--	60	51	56	--	--	--
B1_bis: 0.25<ε<0.89	--	--	--	--	--	--	--
B2: 0.1<ε<=0.25	--	--	63	--	--	--	--
B3: ε<=0.1	--	56	64	55	52	--	--
Enamelled	--	61	71	--	50	--	--

Table 8. Maximum value SC (MPa) vs conform FR for HS in SSV specimens

	4 mm	5 mm	6 mm	8 mm	10 mm	12 mm	15 mm
Float	37	38	31	35	34	34(6%*)	--
B1: ε=0.89	--	--	48	--	--	--	--
B1_bis: 0.25<ε<0.89	--	--	42	--	--	--	--
B2: 0.1<ε<=0.25	--	--	30	--	--	--	--
B3: ε<=0.1	--	31	32	45	37	--	--
Enamelled	--	50(5%)	--	--	42	--	--

Note: (%) incidence value of data in the limit value but NC to FB.
* Sampling with high SC but with "roller effect"

Table 9. Minimum value SC (MPa) vs conform FB for HS in SSV specimens

	4 mm	5 mm	6 mm	8 mm	10 mm	12 mm	15 mm
Float	81	79(<1%)	79	83(1%)	79(<1%)	82(1%)	85
B1: ε=0.89	83	87	81	85	82	--	--
B1_bis: 0.25<ε<0.89	88	--	91(3%)	87	86	--	--
B2: 0.1<ε<=0.25	87	--	82	86(9%)	--	--	--
B3: ε<=0.1	78	88	85(1%)	81(2%)	82(1%)	99(18%)	--
Enamelled	94	94	95	--	91(4%)	--	--

Note: (%) incidence value of data in the limit value but NC to FR.

Table 10. Minimum value SC (MPa) vs conform FB for TT in SSV specimens

In table 8 the data for HS are reported, considering conform and not specimens.

Flexural Bending Strength vs Surface Compression

The data of specimens with SC and FB measurement were considered. All the glass thickness and side in tension were considered (tin, air, coated, un-coated, enamelled) although the SC is measured only at "tin" side, "un-coated" and "un-enamelled" side. Moreover the data were not segregated, considering specimens with both central and edge fracture origin.

All the data of specimens (from 4 mm to 15 mm glass thickness) with recorded SC and FB were considered and the values of SC were recorded and reported in table 9 for heat strengthened glass and table 10 for thermally toughened, in function of glass thickness and type.

In diagrams of figure 4, 5a, b, c, d and 6 the testing value are plotted, showing clearly the type of glass that were tested: heat strengthened and thermally toughened safety glass.

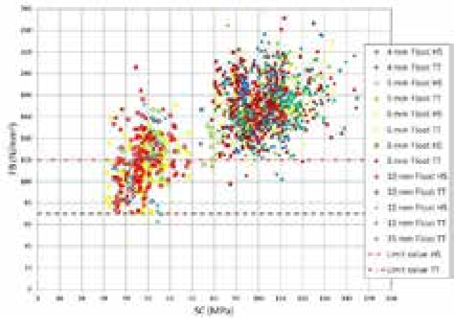

Figure 4. Correlation of surface compressive stress (SC) versus flexural bending (FB) for float glass.

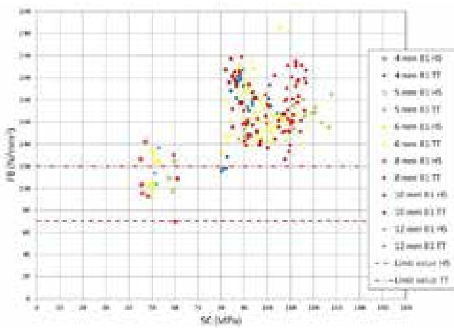

Figure 5a. Correlation of surface compressive stress (SC) versus flexural bending (FB) for B1 coated glass.

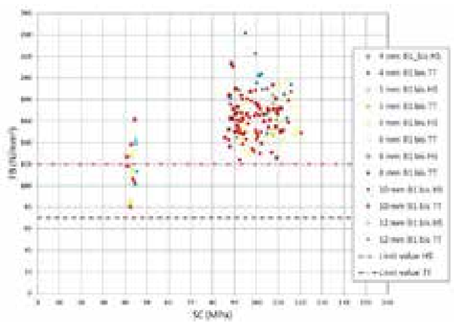

Figure 5b. Correlation of surface compressive stress (SC) versus flexural bending (FB) for B1 bis coated glass.

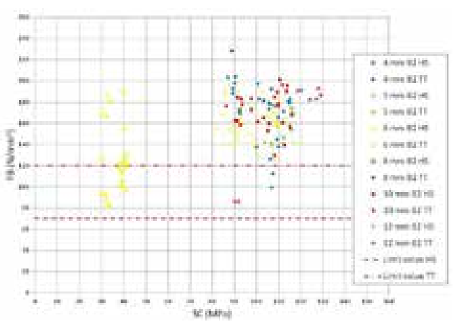

Figure 5c. Correlation of surface compressive stress (SC) versus flexural bending (FB) for B2 coated glass.

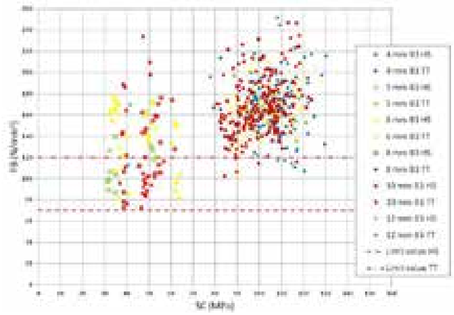

Figure 5d. Correlation of surface compressive stress (SC) versus flexural bending (FB) for B3 coated glass.

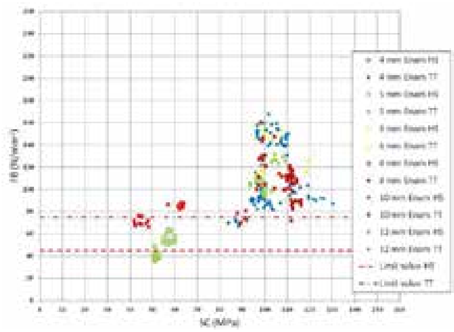

Figure 6. Correlation of surface compressive stress (SC) versus flexural bending (FB) for enamelled glass.

	Limit value SC	4 mm	5 mm	6 mm	8 mm	10 mm	12 mm	15 mm
Float	85	0	0	<1	<1	<1	1	0
B1: ε=0.89	90	0	0	0	0	0	0	--
B1_bis: 0.25<ε<0.89	90	0	--	3	0	0	0	--
B2: 0.1<ε<=0.25	90	0	--	0	9	--	--	--
B3: ε<=0.1	90	0	0	1	1	0	17*	--
Enamelled	95	0	0	0	--	1	--	--

Note: * Sampling with high SC but with "roller effect"

Table 11. Incidence value (%) of NC data for SC (MPa) vs FB in TT with the proposed SC value

Glass Type	FR_HS	FB_HS	FR_TT	FB_TT
	Upper bound	Lower bound	Lower bound	Lower bound
Float	60	35	90	85
B1: ε=0.89	55	50	90	90
B1_bis: 0.25<ε<0.89	45	40	95	90
B2: 0.1<ε<=0.25	55	30	90	90
B3: ε<=0.1	60	40	95	90
Enamelled	60	45	95	95

Table 12. SC value (MPa) respect FR and FB found in SSV testing

The limit value of SC that has to be reached to respect the characteristic strength value of thermally toughened (TT) safety glass can be confirmed to be 85 MPa for float glass and 90 MPa for coated glass; in case of enamelled glass this value should be increased to no less than 95 MPa (Tab. 11).
In the case of heat strengthened glass (HS), the SC value of 35 MPa for float glass can be confirmed. For coated glass the minimum founded SC value was between 30÷50 MPa, whereas for enamelled glass it is 45 MPa (Tab. 12). Some specimens are not conform although the SC is high. As for the SC vs Fragmentation, this is due more to roller effect than non uniformity of SC along the glass thickness.

Conclusions

The elaborated data goes across many years of laboratory tests on different type of glass (heat strengthened and thermally toughened safety, coated and uncoated as enamelled) provided by different producers in Italy and in Europe.
The correlations between SC and FR or FB is accepted at Standard level (see ISO Standard) and it is useful during FPC (Factory Production Control) to evaluate the quality of process by a non destructive procedure. This procedure was defined as the measurements of surface compressive stress on tin side, as prescribed by EN 12150-2:2004 [22] for thermally toughened safety glass, EN 14179-2:2005 [23] for HST glass and EN 1863-2:2004 [24] for heat strengthened glass. The value has to be correlated to fragmentation density (for TT) and to flexural strength (for HS and TT). The not conform specimens were considered too, because they could occur in production and must be detected in the procedure.
The limit value of SC proposed by the authors based on their experimental data are reported in table 12.

References

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