TOWARDS A POTENTIAL STANDARDIZATION OF THE SPLITTING TENSILE-STRENGTH TEST FOR DENSE SHAPED REFRACTORY PRODUCTS

Jean-Pierre Erauw, Isabella Mastroianni, Sandra Abdelouhab, Veronique Lardot and Francis Cambier
Belgian Ceramic Research Centre - Mons – Belgium

ABSTRACT

The splitting tensile-strength test is *a priori* a simple, robust method enabling to approach the true tensile strength of brittle products. It consists in loading diametrally a right-circular cylinder till the fracture strength of the material is reached and the specimen splits along its vertical diameter. The splitting tensile strength is subsequently calculated from the measured maximum load sustained and the dimensions of the specimen. In the current study, the effects of both specimen size and testing conditions (pre-load level, loading rate, use and type of load bearing strips) have been investigated experimentally on two representative dense shaped commercial products. A total of more than 300 splitting tests have been performed. By far the largest effect observed is the one related to the use of bearing strips, the role of which consists in distributing the load applied along the length of the cylinder and in preventing compressive-stress failure near the loading points. All by all, sets of optimum conditions have been identified with regard to repeatability. These will be further evaluated with respect to reproducibility through interlaboratory round robin tests.

INTRODUCTION

Reliable mechanical strength values of refractory products are relevant parameters with respect to the design of durable and safe installations and accurate tensile strength values are in this context of paramount importance. Whereas the cold crushing strength (CCS) method enables to correctly evaluate the compressive strength, bending strength values (i.e. MOR values) are unfortunately not sufficiently representative of the actual tensile strength of the products. Accordingly, a simple, standardized method able to reliably approach the true tensile strength of the products appears still needed.

The splitting tensile-strength test could be such a method. It consists in loading diametrally a right-circular cylinder till the fracture strength of the material is reached and the specimen splits along its vertical diameter. The splitting tensile strength is subsequently calculated from the measured maximum load sustained and the dimensions of the specimen.

The main advantage of this test stems from its ease of implementation. It allows indeed for the use of simple specimen geometry compared to what would be needed for pure tensile test, readily obtained by drilling, and compatible with other, already standardized test methods for refractory products (CCS, density/porosity). Thereupon, the stress state in the specimen during the test is well documented in the literature [1, 2]: the diametral loading induces a uniform tensile stress normal to the loading axis across a rather large part of the anticipated fracture plane whereas relatively high compressive stresses develop around the loading points. This method has for long been applied for the indirect determination of the tensile strength of concrete and has been standardized yet in the civil engineering sector [3].

The current study was accordingly undertaken aiming at identifying the parameters susceptible to influence the test results when applied on dense shaped refractory products, and at assessing sets of robust testing conditions that would guarantee a high level of repeatability.

EXPERIMENTAL

Materials

Two representative dense shaped commercial products have been investigated: a high alumina grade with >75 wt. % alumina, and a magnesia-carbon bonded with carbon content of 10 wt. %. They will be respectively further referred to in this text as HA75 and MC95/10.

Sampling scheme - specimen preparation

The investigated materials were obtained in the form of standard bricks of dimensions 250 x 250 x 75 mm. Sixteen samples were drilled out of each brick according to following scheme (Fig. 1), their axis corresponding to the pressing direction of the brick. After core drilling, the test specimens were brought to the required height by subsequent flat grinding. Care was taken to carefully keep track during the whole preparation and testing steps of the individual position of each specimen within the brick as this has been shown to influence the mechanical resistance values [4].

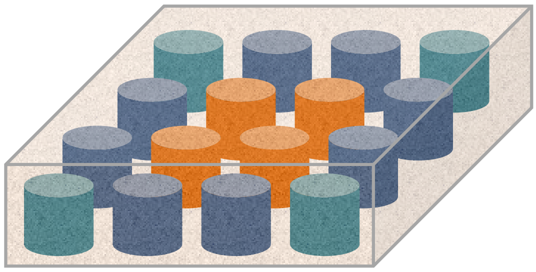

Fig. 1: Schematic of the sampling scheme

After preparation, the specimens were dried to constant mass, cooled to room temperature and stored under a dry atmosphere till tested. When conducting the tests, a single set of parameters is applied by quadrant and replicated at least once.

Splitting tensile-test

The splitting tensile-strength of the specimens was determined on a universal testing machine (Zwick, Z100) equipped with a calibrated load cell of 100 kN capacity. The specimens were placed between the platens of a test jig (Fig. 2) used for the determination of the cold crushing strength, and loaded till failure.

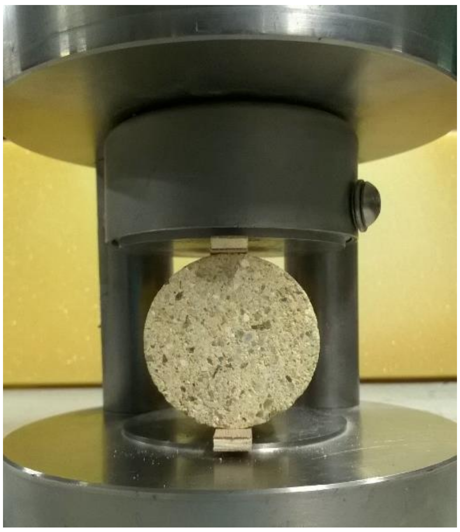

Fig. 2: Test jig for the splitting tensile-test

The splitting tensile-strength was determined as

$$\sigma_{ST} = \frac{2F_{max}}{\pi HD} \quad (1)$$

in which, F_{max} is the maximum force, H, the height of the specimen, taken as the average of four measurements at the extremities of two perpendicular diameters and D, the diameter of the specimen, taken as the average of four measurements on two perpendicular diameters at both extremities of the core. This equation is derived from a linear elastic analysis of the stress field and holds for brittle-elastic materials [2].

Prior to testing, the homogeneity of each series of test specimens has been checked on the basis of their respective geometrical density and Young's modulus. The former was obtained from their mass and dimensions; the latter was evaluated non-destructively from the measured time-of-flight of a 250 kHz ultrasound wave (Krautkramer USD10 NF, probes K0.25G). The observed limited scatter of these results has allowed to consider the whole set of test bars as homogeneous.

Investigated factors

A single specimen geometry has been used throughout this study, namely that of a cylindrical core of nominal diameter 50 mm. This choice is motivated by the fact that this corresponds to the yet standardized geometry of specimens used for the determination of the cold crushing strength (CCS), what might facilitate the future industrial acceptance of the splitting tensile-strength test in the refractory sector. Only the potential effect of the height of the specimen has been investigated in a range corresponding to a 20% tolerance on a nominal height of 50 mm (see Tab. 1).

Tab. 1: Specimen parameter and corresponding levels

Factor	[-1]	[0]	[+1]
Height	40 mm	50 mm	60 mm

The test parameters investigated and their respective low and high levels are summarized in Table 2. Following the requirements of EN 12390-6 [3], hardboard strips (70x15x3,6 mm) were used as packing (i.e. load bearing strip) material. These parameters were combined by pair, in a series of successive 2^2 full factorial design, each set of conditions being replicated 7 times. This resulted in a total of 32 tests per 'campaign'.

Tab. 2: Test parameters and corresponding levels

Factor	[-1]	[+1]
Pre-load	5 N	500 N
Loading rate	0,05 MPa/s	0,15 MPa/s
Packing	No	Yes

Thereupon, a limited number of tests have been performed with a higher pre-load value (2000 N), and a different loading mode corresponding to a constant displacement rate of 1,5 mm/min.

RESULTS AND DISCUSSION

Effect of the specimen dimensions

The average values of splitting tensile-strength obtained on MC95/10 for the three retained specimen dimensions are summarized in Table 3 as a function of testing conditions. They are graphically presented in Fig. 3 together with the individual strength values.

An analysis of variance was subsequently performed on these data. Besides the two investigated factors, this ANOVA was broadened to take into account the potential effects of brick to brick variation and/or location of the specimen within a brick (i.e. centre, corner or side).

Tab. 3: Marginal means as a function of the factors combination

Factor		Mean strength	Coef. of variation
Height	Packing	[MPa]	[%]
-1	-1	4,87 ± 0,50	10,4
0	-1	4,98 ± 0,54	10,8
+1	-1	5,04 ± 0,54	10,8
-1	+1	7,12 ± 0,95	13,3
0	+1	6,60 ± 0,35	5,3
+1	+1	6,59 ± 0,57	8,6

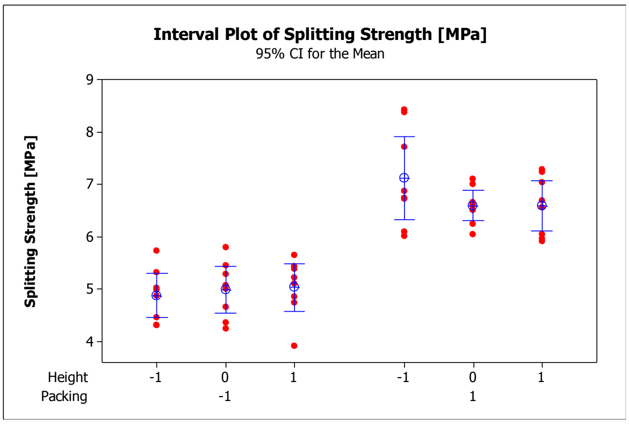

Fig. 3: MC95/10 - Marginal means and individual strength values vs. specimen height and use of load bearing strips

The ANOVA results are summarized in Table 4. The statistically significant effects are highlighted (significance level α=0.05).

Tab. 4: Outcome of the ANOVA for the retained factors

Factor	df	Adj. SS	Adj. MS	F	p
Brick ID	**2**	**4,332**	**2,166**	**7,20**	**0,002**
Location	2	0,038	0.019	0,06	0,939
Height	2	1,240	0,620	2,06	0,141
Packing	**1**	**28,814**	**28,814**	**95,74**	**0,000**
Error	40	12,038	0,301		
Total	47				

As can be seen, within the range tested, the height of the specimen has no significant influence on the resulting splitting tensile-strength value. The packing on the contrary has a major impact on the measured strength. The use of load bearing strips leads to strength values about 35% higher. The location of the specimen within the brick has no impact but one observes a statistically significant effect of the brick itself. This brick to brick variation translates into the non-negligible (but still not uncommon for refractories) dispersion of values around the means.

The same trends were observed for the high alumina grade: no effect of specimen size and a major effect of the use of the load bearing strips leading to a relative increase of strength values of the same order of magnitude. In the case of HA 75, the brick had no influence but a slight effect of the specimen location was noticeable.

As mentioned above, the use of load bearing strips, the role of which consists in distributing the load applied along the length of the cylinder and in preventing compressive-stress failure near the loading points, results in apparent splitting tensile-strength values substantially higher. This is not totally unexpected. Indeed, according to the literature [5,6,7], distribution of the load along a segment of the cylinder leads to a maximum stress value at the center of the test piece, lower than that obtained in a configuration where the same load is concentrated on a generatrix. Accordingly the use of equation 1 for the calculation of the splitting tensile-strength results in an overestimation of the actual rupture strength of the specimen. The dependence of this overestimation on the geometrical characteristics of the bearing strips has been formalized as follows [8]:

$$\sigma_{ST} = \frac{2F_{max}}{\pi HD}(1-\beta^2)^{3/2} \quad (2)$$

with β, the ratio between the width of the interlayer and the diameter of the specimen. In the present case, the use of this correction formula indicates an overestimation of the order of 15%, significantly lower than the one observed experimentally on both MC95/10 and HA75. It was however recently argued [9] than in the case of quasi-brittle materials in which many initial flaws grow and coalesce before the peak load is reached (which might well be the case in the products considered here), this overestimation can be as high as 25%.

Effect of the loading rate

A 'reference' stress rate of 0,15 MPa/s had been chosen, identical to the one used for the MOR testing of dense shaped refractories [10]. The effect of lowering this loading rate to 0,05 MPa/s, following the requirements of EN 12390-6 [3], is illustrated in Fig. 4 for the MC95/10 grade. The corresponding ANOVA results are summarized in Table 5, the statistically significant effects being highlighted (significance level α=0.05).

Tab. 5: Outcome of the ANOVA for the retained factors

Factor	df	Adj. SS	Adj. MS	F	p
Loading rate	**1**	**2,252**	**2,252**	**5,08**	**0,029**
Packing	**1**	**39,628**	**39,628**	**89,42**	**0,000**
Error	45	19,943	0,443		
Total	47				

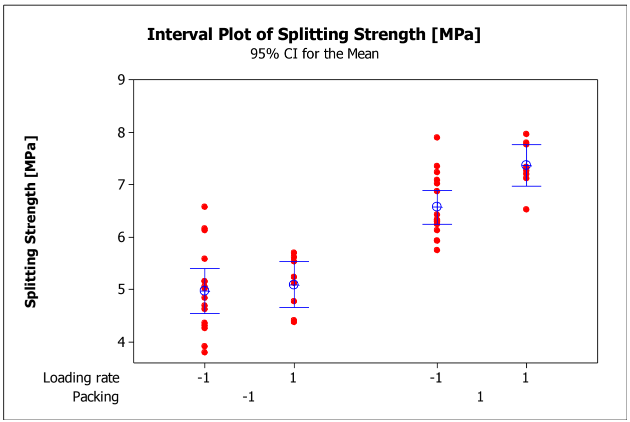

Fig. 4: MC95/10 - Marginal means and individual strength values vs. loading rate and use of load bearing strips

In addition to the effect of the load bearing strips, a positive effect of the loading rate is observed, an increase of the latter resulting in a slight splitting tensile-strength increase. This effect seems more pronounced in the presence of load bearing strips.

Similar effects were observed for the high alumina grade HA75.

Effect of the pre-load level

The splitting tensile-strength values obtained on HA75 for different pre-load levels, with or without packing, are graphically presented in Fig. 5. The test were conducted under the 'reference' stress rate of 0,15 MPa/s. The corresponding ANOVA results are summarized in Table 6 below. The statistically significant effect is highlighted (significance level α=0.05).

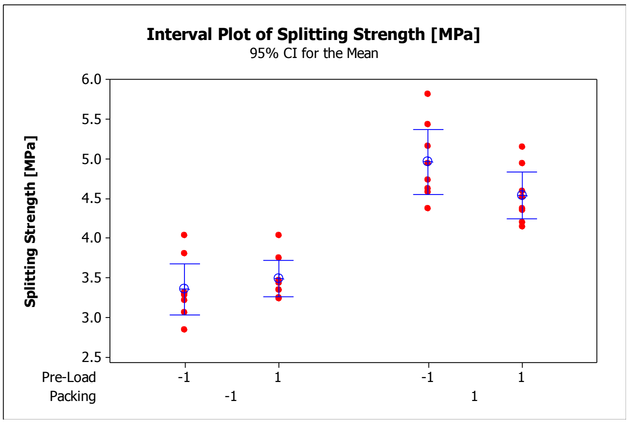

Fig. 5: HA75 - Marginal means and individual strength values vs. pre-load value and use of load bearing strips

Tab. 6: Outcome of the ANOVA for the retained factors

Factor	df	Adj. SS	Adj. MS	F	p
Pre-Load	1	0,176	0,176	1,08	0,307
Packing	**1**	**14,000**	**14,000**	**86,09**	**0,000**
Error	29	4,716	0,163		
Total	31				

The analysis clearly shows that the pre-load factor has, in the range investigated, no significant effect on the results. Only the use of load bearing strips significantly affects the values of the splitting tensile-strength. It should be noted that a higher pre-load is more in line with the recommendation of other testing standards [e.g. ASTM C133] and that its level could be further increased up to 20-30% of the expected rupture strength. The effect of such an additional increase is addressed later (see Preliminary validation).

Preliminary validation

Following test campaign aimed at a 'preliminary validation' of the so far obtained results by replicating a limited number of tests on the high alumina grade in another laboratory. These tests were carried out using a constant displacement rate of 1,5 mm/min and a pre-load level of 2000 N.

All test specimens were prepared by BCRC according to the protocol described above. The marginal means obtained by each laboratory for each set of testing conditions and corresponding 95% confidence interval are graphically presented in Fig. 6 together with the individual strength values.

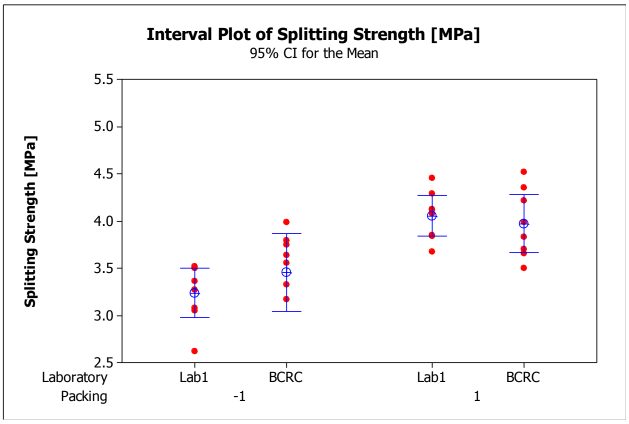

Fig. 6: HA75 - Marginal means and individual strength values vs. laboratory and use of load bearing strips

No statistically significant difference between the results of the two laboratories can be put forward (ANOVA results in Tab. 7), the same major effect of the use of the load bearing strips being also observed by both laboratories.

Tab. 7: Outcome of the ANOVA for the retained factors

Factor	df	Adj. SS	Adj. MS	F	p
Laboratory	1	0,036	0,036	0,26	0,614
Packing	**1**	**3,555**	**3,555**	**25,98**	**0,000**
Error	29	3,968	0,137		
Total	31				

On these tests carried out at a constant displacement speed of 1,5 mm/min, the maximum force supported by the sample is reached on average after 15 ± 1 s. This corresponds to an average stress rate of 0,18 ± 0.02 MPa/s, i.e. a loading rate slightly higher than that used in previous tests (0,15 MPa/s). When using the load bearing strips, this average time increases to 36 ± 2 s. This increase is not compensated for by the higher values of rupture strength, so that the average stress rate in the presence of the load bearing strips is significantly lower, namely 0,10 ± 0,01 MPa/s. However, as mentioned above (see Effect of the loading rate) these differences in stress rate should not affect that much the strength values. This will be further demonstrated hereafter.

One can also stress that the comparison of the mean rupture strength values obtained here with those obtained in previous test campaigns tends once more to indicate the insensitivity of the results with regard to the pre-load used, at least in the range covered by the study : from 5 to 2000 N.

Effect of the nature of the load bearing strips

In order to investigate a possible effect of the nature of the load bearing strips, a series of tests has been carried out in which hardboard has been substituted by cardboard. These tests were carried out on HA75 using as above a pre-load level of 2000 N and two different loading modes: a constant stress rate of 0,15 MPa/s and a constant displacement rate of 1,5 mm/min.
The marginal means obtained for each set of testing conditions and corresponding 95% confidence interval are graphically represented in Fig. 7 together with the individual strength values.

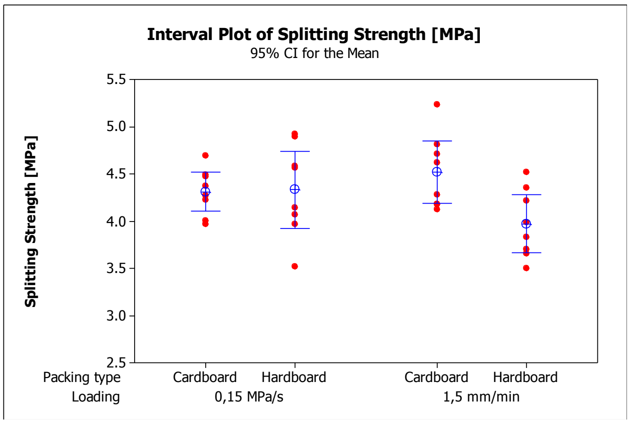

Fig. 7: HA75 - Marginal means and individual strength values vs loading rate/mode and nature of load bearing material

Tab. 8: Outcome of the ANOVA for the retained factors

Factor	df	Adj. SS	Adj. MS	F	p
Loading type	1	0,049	0,049	0,30	0,590
Packing type	1	0,553	0,553	3,35	0,078
Error	29	4,791	0,165		
Total	31				

According to the outcome of the analysis of the variance (Tab. 8), neither the loading mode, nor the nature of the load bearing strips influences the results. It follows that in the present study, irrespective of the nature of the load bearing strip material (hardboard vs. cardboard), the splitting tensile-strength values obtained are similar and in both cases significantly higher than those obtained without the use of these strips.

CONCLUSION

More than 300 tests have been conducted on the retained two grades of dense shaped products. These tests made it possible to investigate all the parameters likely to affect the splitting tensile-strength values. Within the range investigated, none of these parameters seems to affect significantly those values on the exception of the use of load bearing strips. Similarly, no significant effect of the selected set of parameters on the dispersion of the splitting tensile-strength values could be demonstrated. The observed dispersion can largely be attributed to the intrinsic strength dispersion of the material, more or less amplified by the dispersion attributable to the location of the test piece in the brick or to the slight fluctuations in brick-to-brick properties. The detailed understanding of the pronounced effect of the load bearing strips remains at this stage incomplete as the comparison with state-of-the art model(s) let appear a significant discrepancy between experimental values and those that might be expected on the basis of these models. Research efforts in this direction will have to be pursued. However, all by all, sets of optimum conditions have been identified with regard to repeatability. These will be further evaluated with respect to reproducibility through interlaboratory round robin tests and, if proven satisfactory, drafted as potential EN standard and submitted to the relevant CEN Technical Committee.

REFERENCES

[1] Frocht MM. Photoelasticity. John Wiley & Sons Inc., New-York. 1948.
[2] Timoschenko SP, Goodier, JN. Theory of elasticity. Mc Graw-Hill, New-York. 1951.
[3] EN 12390-6: Testing hardened concrete - Part 6: Tensile splitting strength of test specimens. European Committee for Standardization (CEN). 2009.
[4] Erauw JP, Mastroianni I, Lardot V, Cambier F. Investigation of the main factors susceptible to influence the Modulus of Rupture testing results of refractory materials. *Refractories Worldforum*. 2015;7(4):95-103.
[5] Hondros G. The evaluation of Poisson's ratio and the modulus of materials of a low resistance by the Brazilian (indirect tensile) test with particular reference to concrete. *Australian Journal of Applied Science*. 1959;10:243-64.
[6] Markides CF, Kourkoulis SK. The stress field in a standardized brazilian disc: the influence of the loading type acting on the actual contact length. *Rock Mechanics and Rock Engineering*. 2011;45(2):145-158.
[7] Japaridze L. Stress-deformed state of cylindrical specimens during indirect tensile strength testing. *Journal of Rock Mechanics and Geotechnical Engineering*. 2015;7(5):509-518.
[8] Tang T. Effects of load-distributed width on split tension of unnotched and notched cylindrical specimens. *Journal of Testing and Evaluation*. 1994;22(5):401-409.
[9] Rocco C, Guinea GV, Planas J. Elices M. Size effect and boundary conditions in the Brazilian test: Theoretical analysis. *Mater. Struct*. 1999;32:437-444.
[10] EN 993-6: Methods of test for dense shaped refractory products - Part 6: Determination of modulus of rupture at ambient temperature. European Committee for Standardization (CEN). 1995.

ACKNOWLEDGEMENT

This project has received financial support from the Federal Public Service Economy and from the Bureau for Standardisation (NBN) under grant agreement CCN/NBN/PN14B04.
We greatfully thank Dr. Gerhard Urbanek (RHI AG) for providing the dense shaped products investigated here.