Investigation of the Main Factors Susceptible to Influence the Modulus of Rupture Testing Results of Refractory Materials

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Since the adoption of former PRE recommendations as European standards, most testing methods used for the determination of critical mechanical and physical properties of refractory products have not been thoroughly reassessed whereas in the meantime, refractory products have evolved drastically. The adequacy of current testing standards to fulfil today's requirements of the market has become questionable as for instance, current documents lack any statement regarding the accuracy and precision that can be expected from the test methods described. This is particularly true for the test methods used for the determination of the modulus of rupture (MOR) of refractory products. Accordingly, within the framework of the European project ReStaR, "Review and improvement of testing Standards for Refractory products", the current EN standards for the determination of the MOR of dense shaped, insulating (EN 993-6) and monolithic (EN ISO 1927-6) refractory products have been re-evaluated.

In the first stage of this project, as reported here, various factors susceptible to influence the test results have been thoroughly screened through factorial designs of experiments and subsequent analysis of variance in order to identify the most significant ones. These factors have been chosen primarily (but not only) amongst parameters already addressed by the current version of the standards. So far, only the test bar geometry has been found to affect significantly the MOR testing results. Other factors, as for instance the loading rate, have had but a minor, non-significant effect within the range investigated.

1 Introduction

Since the adoption of former PRE recommendations as European standards, most testing methods used for the determination of critical mechanical and physical properties of refractory products have not been thoroughly reassessed whereas in the meantime, refractory products have evolved drastically. The adequacy of current testing standards to fulfil today's requirements of the market has become questionable as for instance, current documents lack any statement regarding the accuracy and precision that can be expected from the test methods described.

This is in particular true for the test methods used for the room temperature determination of the modulus of rupture (MOR). The current version of the EN standard addressing the determination of this characteristic for shaped products, EN 993-6, dates back from 1995, and the equivalent document for unshaped products, EN ISO 1927-6, although more recent, incorporates almost identical requirements in terms of test parameters. However, careful reading of these documents reveals that the described protocols, owing to a lack of precision, leave room for interpretation and can therefore lead to different testing practices which may result in turn, in discrepant results. Accordingly, within the framework of the European project ReStaR, "Review and improvement of testing Standards for Refractory products" (see [1] for details), the current EN standards for the determination of the MOR of refractory products have been re-evaluated.

In the first stage of this project, various parameters susceptible to influence the test results have been thoroughly screened through factorial designs of experiments (DOE) and subsequent analysis of variance (ANOVA) in order to identify the most significant ones. In a second stage, for each type of product considered, a set of maximum three parameters has then been retained

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Tab. 1 Factors and levels chosen for the first DOE on HA75

Factor	Low level [-1]	Central point [0]	High level [+1]
Geometry			
Cross section [mm]	35 x 35	40 x 40	45 x 45
Span [mm]	175	180	185

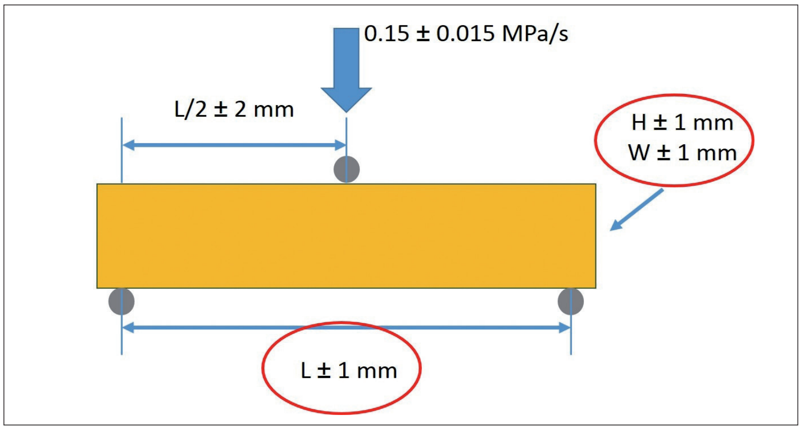

Fig. 1 Schematic of current requirement of EN 993-6 for specimens cut from standard blocks or shapes

for further analysis. Their criticality has been investigated through interlaboratory round robin tests, the details of which being reported elsewhere [2]. On the basis of the outcome of these round robin tests, optimum set of conditions have been identified with regard to repeatability and reproducibility. These will be further evaluated within the last phase of the project and, if proven satisfactory, implemented together with the resulting precision data, in a revised version of current standards to be proposed to the relevant CEN Technical Committee.
In what follows, main results of the preliminary screening exercise are presented.

2 Experimental

2.1 Materials

The study covered dense and insulating shaped products as well as monolithics. The dense shaped refractory used was a high alumina (>75 % Al_2O_3) grade specifically provided by RHI AG for the ReStaR project. This product was delivered as bricks with dimensions 250 x 250 x 75 mm. The insulating shaped refractory was a lightweight insulating grade (>35 % Al_2O_3) from RATH; this product was delivered as standard bricks of dimensions 230 x 114 x 64 mm. The third refractory product tested was a medium cement castable. (>75 % Al_2O_3) produced by Calderys in the framework of this collaborative EU-project. These three products will be respectively further referred to as HA75, LWI35 and MCC75.

2.2 Statistical approach and design of experiments

According to EN 993-6, the MOR is defined as the maximum stress that a prismatic test piece of specified dimensions can withstand when loaded at a constant stress rate in three-point bending. Current version of this standard incorporates primarily requirements in terms of test bar geometry, test jig design (in particular span length) and loading rate. Each of these parameters have reasonably been considered as a factor susceptible to influence the MOR testing results and accordingly thoroughly screened through dedicated factorial DOE and subsequent ANOVA. Depending the case, other potential influencing factors have been considered as the nature of the surface tested (as fired vs. bulk, as cast vs bulk), its location within the brick, …
Choice was made to rely on full factorial designs in order to avoid confounding of main effects and interactions and to run as many replicates as possible taking into account the amount of material available, in order to quantify the random error due to uncontrolled factor.

2.2.1 Dense shaped products (HA75)

A first series of tests was undertaken aiming at assessing the extent of the potential influence of the test bar and test jig geometry. The DOE has consisted in a full factorial design of two factors at two levels (Tab. 1) with the addition of a central point, the tests for each combination of factors being replicated 7 times. The two factors were the cross-section of the bar and the span of the testing jig. The central point of the DOE, corresponds to recommended values in the current version of EN 993-6 for one of the four test bar geometries accepted. The low and high levels are equivalent to adopting on those recommended values, a tolerance 5 times larger than the current ones (Fig. 1). Contrary to the requirement of the standard, the length of the test bars was kept identical to the original brick length (i.e. 230 mm vs 200 mm). A constant stress rate of 0,15 MPa/s was used throughout the experiments.
Current version of EN 993-6 only foresees loading the specimen at constant stress rate. The second series of tests aimed at assessing the potential influence of this constant stress rate value but also of the loading mode itself, by considering the possibility to run MOR tests under constant displacement rate. A full factorial design of two factors at two levels has been adopted (see Tab. 2). The constant stress rate tests have been, as above, replicated 7 times whereas, the constant displacement rate ones could only be replicated 4 times. The chosen stress rate values correspond to the 0,15 MPa/s prescribed value ± 0,05 MPa/s, what corresponds to accepting a tolerance band about three times larger than the 10 % currently accepted. The value of the low (resp. high) level for the constant displacement rate tests has been chosen such that the rupture of the test bars occurs in about the same time interval than when tested under the lower (resp. higher) constant stress rate. Bars of nominal cross section 40 x 40 mm tested using a nominal 180 mm span have been used throughout this second series of experiments.
A final series of tests has been undertaken. On the one hand, it again aimed at quantifying the size effect and thereby confirming a trend observed after the first series of experiments. In practice, two different sam-

ple geometries (i.e. combination of sample cross section and corresponding span) foreseen in the standard have been compared. On the other hand, contrary to what had been done in the two previous series of experiments, as-fired surface have been tested here and the potential effect of their location within the brick (namely upper surface vs. lower surface) has been investigated. A full factorial design of these two factors at two levels has been retained (Tab. 3), each series of tests being replicated 4 times. The stress rate was kept constant and equal to 0,15 MPa/s.

2.2.2 Insulating shaped products (LWI35)

The tests have been performed on cut and ground bars of nominal cross section 40 x 40 mm, using a 180 mm span. Only the potential influence of the loading rate has been evaluated at this stage of the project. Three levels have been tested (Tab. 4); the central point, namely 0,050 MPa/s, corresponds to the current requirement of the European standard; the low and high levels correspond respectively to this nominal value -50 % and +50 %, mimicking a five time larger tolerance on the prescribed stress rate than the current ±10 %. The tests have been replicated 9 times in each group.

2.2.3 Dense unshaped product (MCC75)

In the case of the unshaped product, it had been similarly decided to focus on a single geometry, namely type B, and to keep the span constant and equal to its prescribed value, 180 mm.
Two factors have been retained (Tab. 5): the surface quality of the specimen and the stress rate. The two "surface quality" investigated correspond respectively to the case of bars tested in the as-cast condition and bars free of any potential skin layer, machined from cast blocks of larger dimensions. The stress rate values adopted correspond to the 0,15 MPa/s prescribed value ±0,05 MPa/s. As above, this corresponds to accepting a tolerance band about three times larger. These two factors have been combined in a full factorial design, each set of conditions being replicated, depending the case, 5 or 3 times. The MOR test have been run on specimens fired at 1200 °C.

Tab. 2 Factors and levels chosen for the second DOE on HA75

Factor	Low level [-1]	High level [+1]
Loading conditions		
C[st] Stress Rate [MPa/s]	0,10	0,20
C[st] Displ. Rate [mm/s]	0,18	0,36

Tab. 3 Factors and levels chosen for the third DOE on HA75

Factor	Low level [-1]	High level [+1]
Geometry		
Cross sect. / Span [mm]	25 x 25 / 125	40 x 40 / 180
Tested surfaces		
Type	As fired	
Location	Lower	Upper

Tab. 4 Factors and levels chosen for the DOE on LWI35

Factor	Low level [-1]	Central point [0]	High level [+1]
Loading conditions			
C[st] Stress Rate [MPa/s]	0,025	0,050	0,075

Tab. 5 Factors and levels chosen for the DOE on MCC75

Factor	Low level [-1]	High level [+1]
Loading mode		
C[st] Stress Rate [MPa/s]	0,10	0,20
Test bar nature		
Preparation	As cast	Cut from larger block

2.3 Specimen preparation

2.3.1 Dense shaped product

As mentioned previously, HA75 material was delivered in the form of standard bricks of dimensions 250 x 250 x 75 mm. These bricks have a punch marking in the centre of the lower face whereas on the upper face, an arrow indicates the direction of mould filling.
EN 993-6 standard does not incorporate detailed information or requirements on the preparation of test specimens. It is well known however that surface preparation can have a pronounced effect on flexural strength. In the present case, cut and ground bars have been used. Care was taken to make sure that the saw blade penetrate the material from the side that was going to be tested in tension and test bars of desired dimensions were obtained by subsequent flat grinding of the cut specimens.
In the case of specimens with a cross-section ≥35 x 35 mm, four bars have been taken from each standard brick. Their position with respect to the left edge of the brick (Fig. 2) has been identified as 1, 2, 3 or 4. When combining specimens of 40 x 40 mm and 25 x 25 mm cross-section, up to 6 test bars could be prepared from a single brick; their position has been accordingly identified as 1 to 6. Care was also taken in each case to identify the surfaces of the bars that were perpendicular to the pressing direction.

2.3.2 Insulating shaped products

Two specimens 43 x 43 x 230 mm have been cut in the middle of each LWI35 standard brick. Test bars of nominal dimensions 40 x 40 x 230 mm have subsequently been obtained by grinding the cut specimens. As above, care has been taken to track the faces perpendicular to the pressing direction.

2.3.3 Dense unshaped product

MCC75 test bars have been prepared according to the recommendations provided

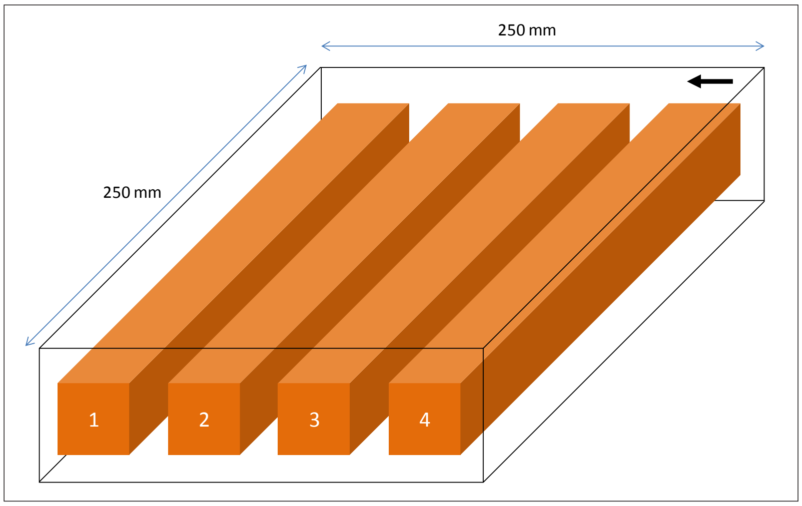

Fig. 2 **Schematic of sampling positions of the test bars in the HA75 bricks; note that the arrow positioned in the upper right corner indicates the mould filling direction**

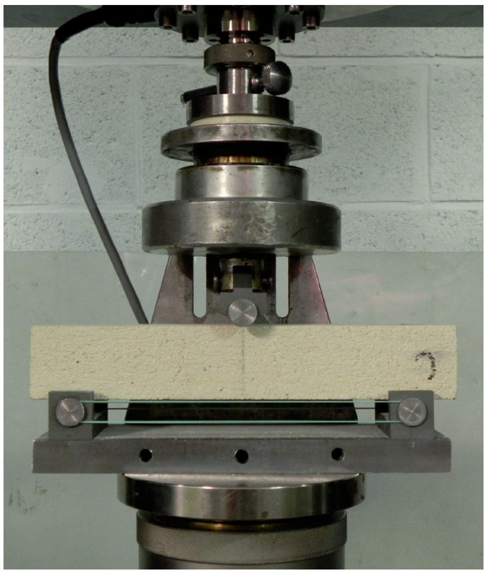

Fig. 3 **Example of test jig used for the 3-pt bending test of dense shaped specimens**

by Calderys. After drying, they have been fired for 5 hours at 1200 °C, the firing cycle following the requirements of EN ISO 1927-5. Besides type B specimens, large blocks of dimensions 250 x 250 x 75 mm were also cast and treated as described above. They have been used for the preparation (by cutting and grinding) of test bars of identical dimensions free of cast surface layers.

2.4 MOR testing

The modulus of rupture (MOR) of the specimens was determined through three-point bending following the requirements of EN 993-6 for the shaped products and EN ISO 1927-6 for the unshaped one, except for the parameters retained as parameters in the DOE described above. Adapted articulated test jigs have been produced (Fig. 3) in order to cover the range of specimen geometries and related spans to be investigated. Prior to testing, the homogeneity of each series of test bars has been checked on the basis of their respective geometrical density and Young's modulus. The former was obtained from their mass and dimensions after drying to constant mass. The latter was measured non-destructively by means of the Impulse Excitation Technique (IET). The natural vibration frequencies of the flexural mode (both Out-of-Plane and In-Plane) and of the longitudinal mode, were experimentally determined. The Young's modulus was calculated using either or both flexural and longitudinal mode according to EN ISO 12680-1.

3 Results and discussion

3.1 Dense shaped products

3.1.1 Homogeneity

Fig. 4 shows the calculated geometrical density values as a function of brick and position within the brick for the 66 test bars used in the two first series of experiments. As can be seen, the average geometrical density appears overall homogeneous between the different bricks. This density seems slightly affected by the position within the brick; this effect can probably be related to the fact that the test bars have been cut perpendicular to the mould filling direction. Identical behaviour was obtained for the additional 20 test bars used in the third series of experiments. Similarly, Fig. 5 reports the corresponding individual Young's modulus values. As can be seen, some variability exists between the different

Brick number	Position 1	2	3	4	Avg	StDev
32	2.72	2.74	2.74	2.72	2.73	0.01
33	2.70	2.71	2.71	2.70	2.71	0.01
35	2.70	2.72	2.72	2.72	2.71	0.01
36	2.70	2.72	2.72	2.71	2.71	0.01
38	2.70	2.71	2.72	2.71	2.71	0.01
39	2.70	2.71	2.71	2.70	2.71	0.01
42	2.69	2.72	2.72	2.71	2.71	0.01
43	2.69	2.71	2.73	2.73	2.71	0.02
44	2.72	2.73	2.73	2.73	2.73	0.01
45	2.71		2.71	2.70	2.71	0.01
46	2.70		2.73	2.72	2.72	0.01
47	2.70		2.71	2.69	2.70	0.01
48	2.70	2.71	2.71	2.70	2.70	0.01
49	2.70	2.72	2.72	2.72	2.71	0.01
50	2.70		2.72	2.72	2.71	0.01
51	2.69		2.72	2.75	2.72	0.03
52	2.71		2.73	2.71	2.72	0.01
91	2.69	2.71	2.71	2.69	2.70	0.01
Avg	2.70	2.72	2.72	2.71		
StDev	0.01	0.01	0.01	0.01		

35x35
40x40
45x45

Fig. 4 **Calculated geometrical density (in g/cm³) of test bars as a function of HA75 brick and position within the brick**

Brick number	Position 1	2	3	4	Avg	StDev
32	55	47	48	49	50	3
33	42	41	43	42	42	1
35	43	46	46	49	46	3
36	41	44	45	42	43	2
38	41	43	43	42	42	1
39	42	41	43	41	42	1
42	40	46		42	43	3
43	42	54	47	59	50	8
44	52	62	58	57	57	4
45	44		46	51	47	3
46	45		47	47	46	1
47	45		41	47	44	3
48	46	49	46	45	46	2
49	43	48	44	44	45	2
50	42			46	44	3
51	54		56	57	55	2
52	51		51	47	50	2
91	38	41	44	42	41	3
Avg	45	47	47	47		
StDev	5	6	5	6		

35x35
40x40
45x45

Fig. 5 **Experimental Young’s modulus value (in GPa) of test bars as a function of HA75 brick and position within the brick**

bricks, but no particular pattern depending on the position within the brick appears. The overall acceptable scatter allows us to consider the whole set of test bars as homogeneous. Nevertheless, in each series of experiments, care has been taken to sample the different groups of test bars over the different bricks and positions.

3.1.2 First series of experiments

The marginal means corresponding to the five individual combinations of factors are given in Tab. 6.

To assess the significance of the observed differences between these means, ANOVA has been performed, the results of which are summarized in Tab. 7. The statistically significant effects are highlighted (significance level α = 0,05). The corresponding Pareto chart of the effects is shown in Fig. 6.

Two factors appear to have a statistically significant effect: the cross section of the specimen and the first order interaction between the cross section and the span. The effect of cross section is negative which implies that with increasing cross section, the response (modulus of rupture) tends to decrease. In practise, a 10 % decrease of MOR is observed when increasing the cross section from 35 x 35 mm (mean MOR = 13,15 ± 1,50 MPa) to 45 x 45 mm (mean MOR = 11,91 ± 0,66 MPa). This decrease is however not much larger than the calculated 6% uncertainty band on the MOR mean values. It can therefore be reasonably assumed that for the currently prescribed smaller tolerance on bar dimensions (i.e. ±1 mm) the potential variability of strength caused by ill-controlled sample dimensions will not be significant. The span itself has apparently no effect on the obtained values of the modulus of rupture. Finally, the negative effect of the interaction between the two factors studied is less straightforward to explain.

3.1.3 Second series of experiments

The marginal means corresponding to the individual combinations of factors are provided in Tab. 8. The Pareto chart of effects, as resulting from the ANOVA of this set of data is given in Fig. 7.

As can be inferred from these data, nor the loading rate, nor the loading mode, nor their interaction has a statistically significant effect (significance level α = 0,05) on the resulting values of modulus of rupture, at least when taken at the levels investigated here. This implies that the current tolerance on the stress rate, much smaller than that adopted in this research, although satisfactory, could eventually be increased without significant impact on the test results. It also suggests that loading the test bars under constant displacement rate could eventually be accepted in the revised version of

Tab. 6 **First DOE on HA75 – Marginal means as a function of the factors combination**

Factor		Modulus of rupture [MPa]				
Cross Section	**Span**	**N**	**Mean**	**StDev**	**-95% Conf. limit**	**+95% Conf. limit**
[-1]	[-1]	8	12,64	0,90	11,91	13,37
[-1]	[+1]	8	13,66	1,85	12,93	14,39
[0]	[0]	8	12.56	0.57	11.83	13.29
[+1]	[-1]	8	12,25	0,66	11,52	12,98
[+1]	[+1]	8	11,56	0,45	10,83	12,29

Note : 95% CIs based on mean sq. error 1,038

Tab. 7 First DOE on HA75 – ANOVA results

Factor	**df**	**Sum Sq.**	**Mean Sq.**	**F**	**p**
Cross section (A)	1	12,375	12,375	11,924	**0,001**
Span (B)	1	0,228	0,228	0,220	0,642
A * B	1	5,865	5,865	5,651	**0,023**
Error	36	37,363	1,038		
Total	39	55,831			

Tab. 8 **Second DOE on HA75 – Marginal means as a function of the factors combination**

Factor		Modulus of rupture [MPa]				
Loading rate	**Loading mode**	**N**	**Mean**	**StDev**	**-95% Conf. limit**	**+95% Conf. limit**
[-1]	[-1]	5	12,56	1,18	11,75	13,37
[-1]	[+1]	8	12,23	1,03	11,59	12,86
[+1]	[-1]	5	12,64	0,73	11,83	13,45
[+1]	[+1]	8	12,51	0,46	11,88	13,15

Note : 95% CIs based on mean sq. error 0,754

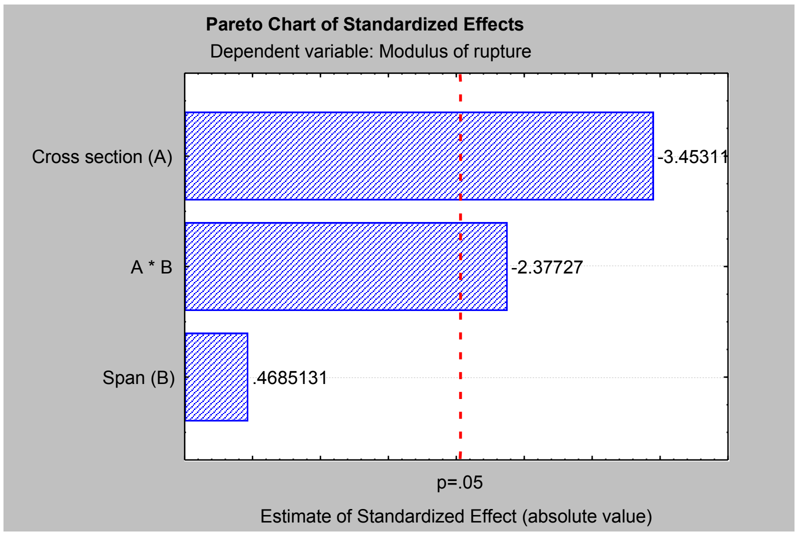

Fig. 6 **Pareto chart of effects in the first DOE on HA75**

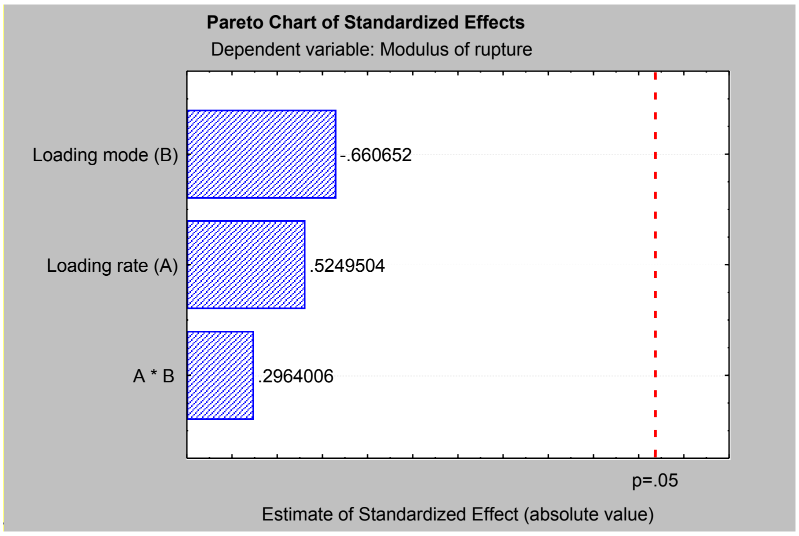

Fig. 7 Pareto chart of effects in the second DOE on HA75

the standard depending on the equipment available in the testing laboratory.

3.1.4 Third series of experiments

The marginal means corresponding to the individual combinations of factors are given in Tab. 9 and graphically represented in Fig. 8. The outcome of the analysis of variance is summarized in the Pareto chart given in Fig. 9.

According to the analysis, the specimen size (i.e. the cross section itself and corresponding span) is the only relevant effect. This negative effect, implying that the MOR values tend to decrease when increasing the volume of the specimen stressed in tension, validates what has already been observed during the first set of experiments. This effect is in the present case, far from being negligible. The overall mean MOR value obtained for the larger samples (i.e. pooling all data for the 40 x 40 mm test bars, neglecting the non-significant effect of surface location) is 12,53 ± 1,26 MPa; this is about 20 % lower than the value obtained for smaller 25 x 25 mm specimens (namely 15,74 ± 1,24 MPa).

Such a dependency of the strength on the component size is however not totally unexpected. The strength of a brittle material, as it is the case for the refractory products, is not a deterministic quantity but will largely vary depending on the flaw population within the material. As a corollary, the larger the specimen, the "lower" the strength as the chance of having a more severe flaw becomes larger as well. There will be accordingly an inherent statistical scatter in the strength test results. This variability can be quantified and modeled using Weibull statistics, provided a sufficient number of data are generated (i.e. a sufficient number of specimens tested). Ultimately, this Weibull statistic will enable to predict this dependency on size [3] according to:

$$\frac{\overline{\sigma_1}}{\overline{\sigma_2}} = \left(\frac{V_{eff2}}{V_{eff1}}\right)^{1/m} \qquad (1)$$

where σ_1 and σ_2 are the mean strength values of specimens of type 1 and 2 (which may have different sizes and stress distributions), V_{eff1} and V_{eff2}, the respective effective volumes of each type, and m, the Weibull modulus. The above relation assumes a two-parameter Weibull distribution as well as a unimodal and homogeneously distributed flaw population. The effective volume is often express as $L_f.V$, with L_f a so-called load factor which depends on the loading configuration and Weibull modulus, and V, the total volume of the specimen under stress (i.e. for bending tests, the total volume within the outer loading points). In the present case, all specimens having been tested in 3-pt bending, the above relation reduces to:

$$\frac{\overline{\sigma_{40\cdot40}}}{\overline{\sigma_{25\cdot25}}} = \left(\frac{V_{25\cdot25}}{V_{40\cdot40}}\right)^{1/m} = \left(\frac{25\cdot25\cdot125}{40\cdot40\cdot180}\right)^{1/m} = 0{,}27^{\,1/m} \qquad (2)$$

Tab. 9 Third DOE on HA75 – Marginal means as a function of the factors combination

Factor		Modulus of rupture [MPa]				
Surface position	**Specimen size**	**N**	**Mean**	**StDev**	**-95% Conf. limit**	**+95% Conf. limit**
[-1]	[-1]	5	15,27	1,09	14,08	16,46
[-1]	[+1]	5	12,75	1,21	11,55	13,94
[+1]	[-1]	5	16,22	1,31	15,02	17,41
[+1]	[+1]	5	12,31	1,41	11,12	13,50

Note : 95% CIs based on mean sq. error 1,586

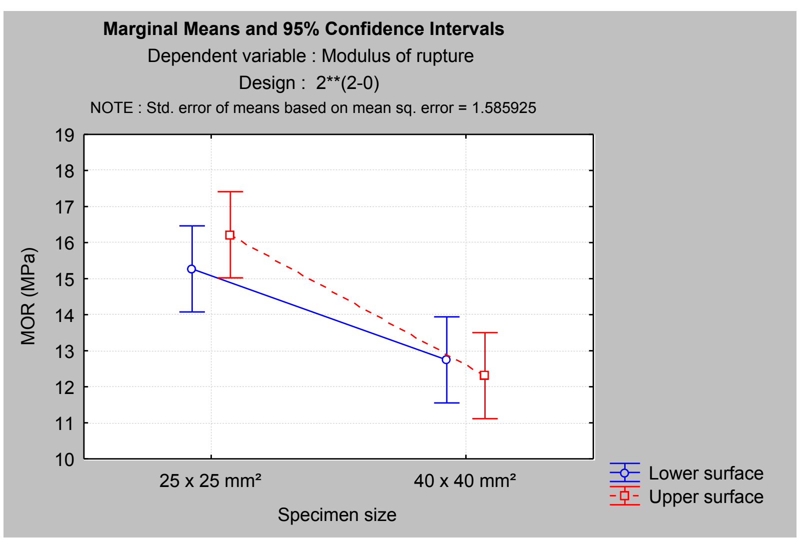

Fig. 8 Effect's plot corresponding to the outcome of the second DOE on HA75

An unbiased estimate of the Weibull modulus (m = 9,8) has been calculated following the maximum likelihood method as described in EN 843-5. Using this estimate, a ratio of 0,88 between the mean strength value of the larger specimens and that of the smaller ones is predicted. In practice, a somewhat larger decrease of strength (20 % instead of the predicted 12 %) is observed.

As already said, current version of EN 993-6 allows the use of four different types of sample geometry. In view of the above, it seems necessary to make clear in the revised version that considerable care must be used when comparing the results of different determinations of the MOR, in particular when comparing numerical results obtained by testing specimens of different geometry.

The second factor investigated in this third series, the location of the tested surface, has no statistically significant effect on the MOR. As far as HA75 material is concerned, upper and lower surfaces of the bricks lead to equivalent MOR results. Thereupon, it is worth pointing out that the average mean strength obtained when testing as-fired surfaces, 12,53 ± 1,26 MPa (pooled results for the 40 x 40 mm geometry) is equivalent to that obtained when testing bulk (i.e. inner) surfaces, 12,46 ± 0,83 MPa (pooled results from the second series of experiments on 40 x 40 mm test bars).

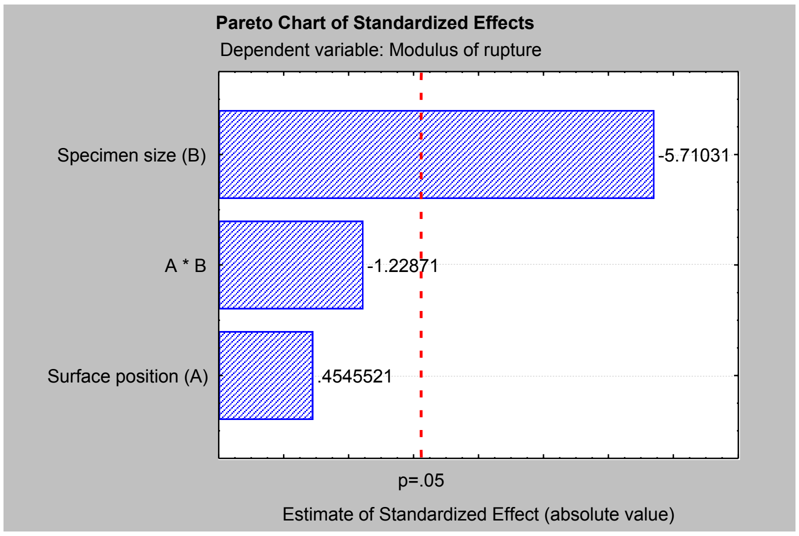

Fig. 9 Pareto chart of effects in the third DOE on HA75

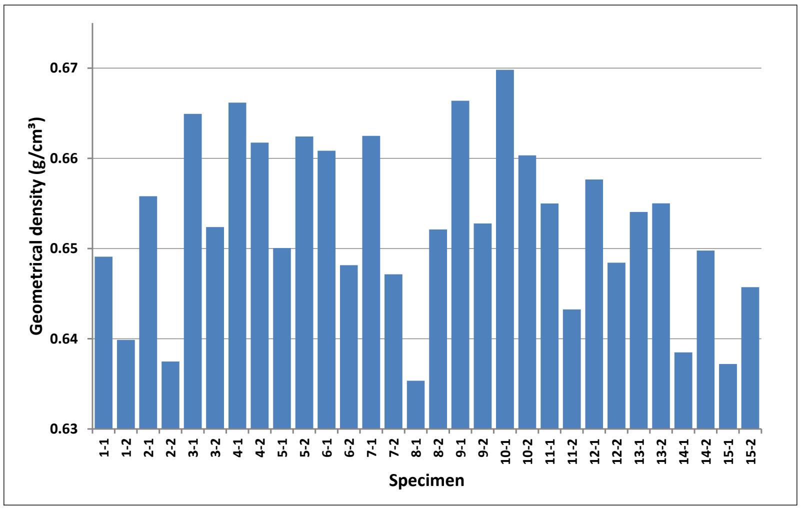

Fig. 10 Geometrical density values of individual test bars

3.2 Insulating products

The individual geometrical bulk density values of the 30 test bars are presented graphically in Fig. 10. The overall average amounts 0,65 ± 0,01 g/cm³. Despite this overall homogeneity, close examination of the figure shows that within each brick, one bar presents systematically a slightly higher density than the other, suggesting a possible density gradient within each brick.

The Young's modulus of each bar has been determined by IET. The overall mean value obtained is 1,47 ± 0,18 GPa. The observed limited scatter allows again to consider the whole set of test bars as homogeneous. In the present case, the sampling of the three groups of ten bars used in the MOR tests has been done on the basis of the above data.

The mean MOR values and corresponding standard deviation obtained for each level of the factor retained here, are given in Tab. 10 and graphically represented in Fig. 11.

According to the one-way ANOVA performed on the experimental data, the loading rate, within the range covered by the experiments, has no statistically significant effect on the measured modulus of rupture (significance level α = 0,05). The apparent slight increase of mean MOR value with increasing stress rate, although not totally unrealistic, remains marginal with respect to the confidence band of the calculated average values.

As mentioned previously, this suggests that the current tolerance on this parameter, as stated in the standard, seems perfectly appropriate, especially recalling that it allows a stress rate range much smaller than what has been investigated here.

3.3 Dense unshaped products

The marginal means corresponding to the four individual combinations of factors studied are given in Tab. 11. As can be inferred from the outcome of the ANOVA (Fig. 12), in the case of the MCC75 used here, nor the factors retained nor their first order interaction have a statistically significant effect on the modulus of rupture.

4 Conclusion

Several factors have been tested on dense shaped products: the test bar geometry, loading rate, loading mode, position of the surface tested. At this point, only the bar geometry has been shown to have significant impact on the resulting MOR values. It was demonstrated that to some an extent, the magnitude of this effect could be esti-

Tab. 10 DOE on LWI35 – Marginal means as a function of the factors combination

Factor	Modulus of Rupture [MPa]				
Loading rate	N	Mean	StDev	-95% Conf. limit	+95% Conf. limit
[-1]	10	1,21	0,09	1,15	1,27
[0]	10	1,26	0,15	1,15	1,37
[+1]	10	1,29	0,10	1,22	1,36

Note: 95% CIs based on mean sq. error 0,013

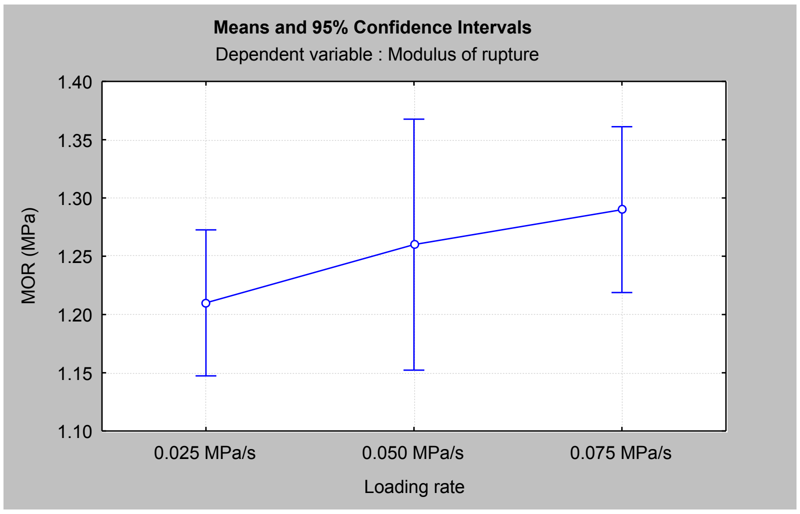

Fig. 11 Effect's plot corresponding to the outcome of the DOE on LWI35

Tab. 11 DOE on MCC75 – Marginal means as a function of the factors combination

Factor		Modulus of rupture [MPa]				
Surface type	Loading rate	N	Mean	StDev	-95% Conf. limit	+95% Conf. limit
[-1]	[-1]	4	10,28	0,55	9,62	10,93
[-1]	[+1]	4	10,99	0,71	10,33	11,64
[+1]	[-1]	6	10,71	0,66	10,18	11,25
[+1]	[+1]	6	10,70	0,56	10,17	11,24

Note: 95% CIs based on mean sq. error 0,382

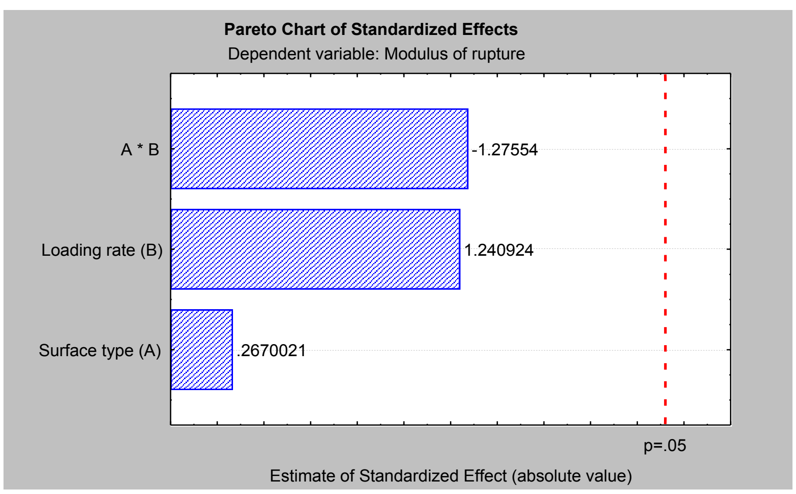

Fig. 12 Pareto chart of effects in the DOE on MCC75

mated using Weibull theory. On the insulating products, only the effect of the loading rate has been evaluated and appeared to have, within the range covered by the experiments, no statistically significant effect on the measured MOR. Finally, two factors have been considered in the case of the monolithic product, the loading rate and the quality of the surface tested. It has been observed that nor the factors nor their first order interaction have a statistically significant effect on the MOR when taken at the chosen levels.

In none of the two latter products, the effect of the specimen size has been investigated in this stage of the project. Taking into account the observations on the HA75 material, it has been decided to consider this factor in the case of shaped insulating and unshaped products in forthcoming experiments and to quantify this effect in those products as well.

Whatever the material considered here, the loading rate did not affect the MOR value obtained. This clearly suggests that the current tolerances on this parameter, much smaller than those adopted in this research, is satisfactory but could eventually be increased without significant impact on the MOR test results. This factor will accordingly be considered in forthcoming interlaboratory comparison, in order to validate the current observation and above conclusion.

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References

[1] E. Brochen, P. Quirmbach, A. Volckaert. ReStaR – Concerted effort in the European refractory sector to consolidate and make EN testing standards future-proof. refractories WORLDFORUM **7** (2015) [3] 85-86.

[2] J.P. Erauw, I. Mastroianni, V. Lardot and F. Cambier. Reappraisal of current standard testing methods for the determination of the modulus of rupture (MOR) of refractory products (Proceedings of UNITECR 2015).

[3] D. Davies, The Statistical Approach to Engineering Design in Ceramics, Proc. Br. Ceram. Soc. (1973) [22] 429–452.