Unshaped Refractories – Selling and Installation Globally – Development and Testing Locally?

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Producers, customers, engineering and installing companies are in urgent need of unified, internationally valid test procedures for obtaining figures and values of properties of unshaped refractories. Without such standards no "true" data sheets of existing products and no real worldwide comparison of product data can be made. Decisions regarding acceptance or refusal of deliveries to job sites depend on applied standards and can often end in unpleasant, time-consuming, costly and redundant discussions and trouble between involved parties. And last but not least, international standards are important, too, for improving and developing products. This paper is intended as a contribution to the objective of agreeing internationally valid standards as quickly as possible.

1 Review and requirements

The author has been a member of national and international bodies for the standardisation of unshaped refractories for more than 35 years. In 1999 [1] he reported on EN 1402 and invited all involved parties to make use of the new standards. The result was that today this standard is applied in Europe by users and producers.

During the last International Colloquium on Refractories in 2008 in Aachen, a report was given by *Krause* and *Krebs* [2] about results obtained by testing a castable according to different international standards. Relevant standards from Australia, United States, Japan/India and Europe were applied. Tab. 1 shows the results as a reminder.

Tab. 1 Results of investigations on a castable according to different standards after firing at 1000 °C [1]

Test specimen	Sizes [mm]	Volume [dm³]	PLC [%]	MOR [N/mm²]	CCS [N/mm²]
Jap./Ind. bar	160 x 40 x 40	0,256	-0,36	**20,9**	**130**
EN shape A/10h	230 x 114 x 64	1,678	-0,34	16,8	115
US cube (CCS only)	51 x 51 x 51	**0,133**	-0,36	n.d.	115
EN shape B	230 x 64 x 54	0,795	-0,32	16,7	112
US bar	228 x 51 x 51	0,593	**-0,31**	17,78	111
EN shape A/5h	230 x 114 x 64	1,678	-0,33	14,9	109
AS bar	230 x 115 x 75	**1,984**	**-0,37**	**11,2**	**97**
Average		**1,017**	**-0,34**	**16,4**	**113**

Regarding the modulus of rupture (MOR) and cold crushing strength (CCS) there was a tendency for test specimens with square cross sections and square compression areas to show "good" or "better" results. Shape B of EN 1402, with test height = 54 mm, test width = 64 mm and a square compression area, showed CCS results near to the Japanese bar and American cube.

Big differences can be recognized in the column "Volume" of test specimens. The volume of an Australian bar is about 14 times greater than that of the US cube (used for CCS only). Fig. 1 shows the significant differences in the sizes of the investigated test specimens.

In the end, all refractory specialists know very well that there is no correlation which could end in mathematical rules like the

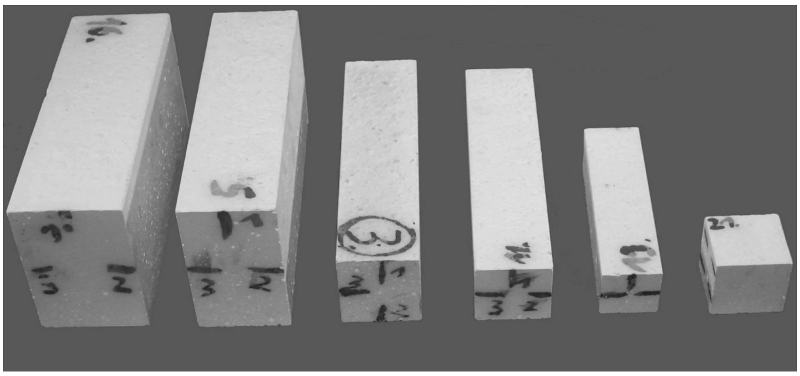

Fig. 1 Test specimens used in [2], from left: Australian bar, EN shape A, EN shape B, US bar (normally for MOR only), Indian/Japanese bar, US cube (for CCS only)

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Japanese-CCS = factor x times EN-CCS
or
AS-MOR = factor y times ASTM-MOR.

During the laboratory tests [2], many test specimens were prepared and many PLC (permanent linear change), MOR and CCS tests were carried out. Due to the large number of cases, an evaluation of the deviation of the averages for the relevant properties would seem permissible. This is done in Tab. 2.

The biggest test specimen – the Australian one – had the lowest strength values (and highest shrinkage), whereas the highest figures for MOR and CCS were determined on the Japanese/Indian shape. Due to the fact that the permanent linear change (PLC) generally varied within a small range, no great significance should be attached to the values for this parameter. However, test specimen volume is an important aspect, especially in relation to material quantities which have to be handled in laboratories. An "ideal volume" can be estimated to be approximately one litre.

Based on these findings, requirements for a universal standard can be postulated. For this an "international test specimen" has to be found first. Such a universally valid test specimen is the most important part of a globally valid standard. Without a test specimen, valid all over the world, no classification is possible and no comparable properties can be determined.

Based on experience it can be said that cutting off a test specimen to determine strength is not necessary, often wrong. A proper shape should allow the determination of as many properties as possible. This includes bulk density BD, porosity, PLC, MOR and CCS and should allow the drilling of additional test specimens for hot tests like refractoriness under load (RUL) or creep in compression (CIC).

Based on Fig. 2 the requirements for a universal standard can be formulated as follows:

- Given the results mentioned above [2], the cross section should be a square one.
- The load area during CCS test should be square too.
- The test-specimen volume underneath the pressing plates in CCS devices should be a cube during the test.
- For additional investigations such as RUL and CIC, cylindrical test specimens (H = 50 mm, Ø = 50 mm) must be drilled out of the larger test piece. Therefore the testing height (which is the preparation width) should be at least 50 mm for drilling out cylindrical test specimens. Additionally there is a need to compensate for shrinkage (after pre-firing) and loss of material when grinding cylinders. Experience shows that the minimum height must be 54 mm.
- The testing width should include 50 mm for drilling cylinders + 2 x 3 mm space for the drill plus residual material for fixing during drilling. A width of 64 mm is good and practicable.
- The length of the test specimen should be 230 mm, which is the relevant standard length in Europe, Australia and America. Finally, a MOR test with supports at a distance of 180 mm should be possible without any problems.
- The total geometry should allow the preparation of test specimens from coarser unshaped refractories without any problem. 2-inch cubes or bars having a cross section of only 40 mm x 40 mm are not suitable for fulfilling such requirements.
- Since the final test specimen must be valid for rammings, plastics, gunnings etc., the testing direction has to be rectangular to the direction of preparation by ramming, vibration, gunning, wet shotcreting, self-flowing etc. in every case.

Finally it should not be forgotten that a large test specimen is also necessary for e.g. determining the properties of "light" LW castables, for determining thermal conductivity etc. This can be realised easily with shape A according to EN 1402.

Tab. 2 Deviations of results on test specimens from averages according to [2]

	Deviation from average [%]			
Test specimen	**Volume**	**MOR**	**CCS**	**PLC**
AS bar	**+95,1**	**-31,7**	**-14,2**	**+8,8**
EN shape A/5h	+65	-9,2	-3,5	-0,3
EN shape B	-21,8	+1,8	+0,9	-5,9
EN shape A/10h	+65	+2,4	+2,7	0
EN shape C	-7,4			
US bar	-41,7	+7,9	-1,8	**-8,8**
Jap bar	-74,8	**+27,4**	**+15,0**	+5,9
US cube	**-86,9**	n.d.	+1,8	+5,9
Average	**1,017 dm³**	**-0,34%**	**16,4 N/mm²**	**113 N/mm²**

2 Additional experiments

All the above facts indicate that test specimens of shape B (54 mm x 64 mm x 230 mm) or shape C (64 mm x 64 mm x 230 mm) of EN 1402 could fulfil all the needs. Within the study [2] unfortunately only shape A and shape B were investigated and not shape C.

As a follow up, results of investigations will be shown, where MOR and CCS were determined on test specimens of shape B and C. Five different commercially available refractory castables were tested:

- LW castable based on LW chamotte, BD = 1,4 g/cm³, 40 % Al_2O_3, 12,5 % CaO
- Regular castable (RC) based on chamotte, BD = 2,0 g/cm³, 40 % Al_2O_3, 11 % CaO
- LCC based on low iron chamotte, BD = 2,3 g/cm³, 52 % Al_2O_3, 2,4 % CaO

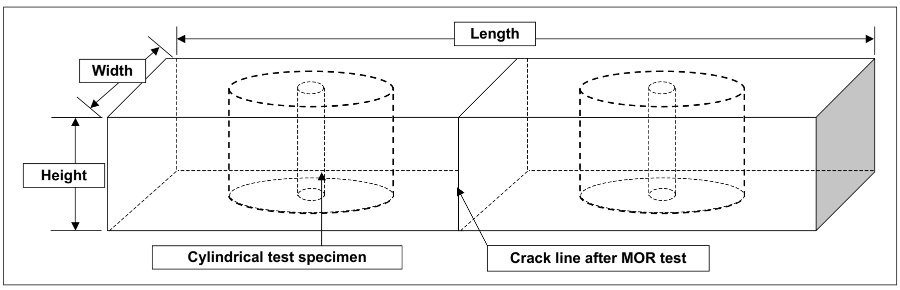

Fig. 2 Demands on a suitable test specimen, especially designed for drilling cylindrical test pieces (H = 50 mm, Ø = 50 mm)

Tab. 3 MOR and CCS of different castables after drying and after firing at 1000 °C according to EN 1402 as a function of test-specimen shape

Material type	LW castable		RC castable		LCC, low iron		LCC, andalusite		ULCC, corundum	
Test piece shape	B	C	B	C	B	C	B	C	B	C
After 110 °C										
MOR [MPa]	**4,3**	**4**	**9,1**	**12,1**	**18,6**	**16,5**	**10,8**	**12**	**3,9**	**4,2**
Standard deviation	0,3	0,2	0,2	2,9	1,3	0,6	0,5	0,4	0,1	0,2
CCS [MPa]	**30**	**26**	**83**	**86**	**125**	**126**	**67**	**62**	**32**	**21**
Standard deviation	0,3	0,5	6,0	2,2	4,6	5,1	2,4	3,0	1,4	1,5
After 1000 °C / 5 h										
MOR [MPa]	**1,9**	**1,7**	**3,2**	**3,2**	**15,4**	**14,8**	**13**	**13,5**	**4,9**	**5,5**
Standard deviation	0,2	0,1	0,2	0,1	0,2	1,3	0,6	1,1	0,1	0,5
CCS [MPa]	**15**	**13**	**32**	**30**	**100**	**96**	**78**	**77**	**30**	**23**
Standard deviation	0,4	0,6	1,1	4,0	1,8	4,4	2,5	5,0	1,4	1,1

- LCC based on andalusite, BD = 2,6 g/cm³, 62 % Al_2O_3, 1,4 % CaO
- ULCC based on tabular alumina, BD = 3,2 g/cm³, 99 % Al_2O_3, 0,4 % CaO.

Three test specimens were prepared carefully from each castable and each shape. The results after drying at 110 °C (24 h) and after firing at 1000 °C (5 h) are shown in Tab. 3.

Each MOR value is the average of three tests, each CCS value of six tests. The following facts can be concluded:

- It can be seen from all the strength tests that shape C has a higher strength in only 20 % of all cases. This fact does not support the theory that square cross sections and cube volumes automatically result in better or higher strengths.
- C has a lower standard deviation shape value in only 20 % of all cases.
- The differences between MOR and CCS of shapes B und C seem to be smaller after pre-firing for each type of castable. This can be traced to the elimination of the influences of the quantities of residual CAH phases after drying.

All the values are close; so based on these results both test specimens could be recommended as an international standard. However, if the findings of the previous investigation [2] are taken into consideration, shape C should be preferred.

3 Importance of testing equipment

It is also very important that laboratories should be well equipped to fulfil the requirements of the standards. This importance will be demonstrated by the following example.

A user of a castable refractory (type RC, based on low iron flint clay) undertook an incoming inspection on each delivery to his laboratory. He vibrated a shape A test specimen. Cylindrical test specimens were drilled out and ground after curing, and properties like BD and CCS were determined after drying at 110 °C / 24 h. However, the customer's results could never be verified in the producer's laboratory. In order to find the reasons for these discrepancies, comparison tests were carried out at the laboratories of the producer, customer and two refractory institutes. The specimens for testing were prepared by the producer according to the drawing shown in Fig. 3, and BD was also determined by the producer. Afterwards, these samples were sent to the participant parties.

CCS tests were made at the same time in order to avoid any external influences (different storage time etc.). The results are shown in Fig. 4, which plots individual and average values for each laboratory.

The diagram demonstrates two aspects:

- The fact is that the results of lab C are out of line with the other three laboratories. Their results are close together, and the averages are in a range of 3 MPa. The reason for this was that lab C was not well equipped to fulfil the needs of any refractory standard – neither for shaped nor for unshaped refractories. No correct pressing devices were used as prescribed. Additionally, continuously increasing stress for supporting the load onto the test specimens was not possible in the press.
- On the other hand, it is remarkable that in each laboratory test specimen No. 5 always has the lowest value. The exception is lab C, where No. 1 showed the lowest CCS value. Nevertheless, here as well, No. 5 is significantly below the average value. The main reason can be recognized from the evaluation of Tab. 4, where all the bulk densities are listed. Obviously No. 5 has the lowest BD in all cases and this is the explanation for the above mentioned phenomenon. Number 5 was taken out of the centre of shape A, whereas the other four were taken out of corner areas. This shows that compaction of the castable in the centre area is not as efficient as in other areas.

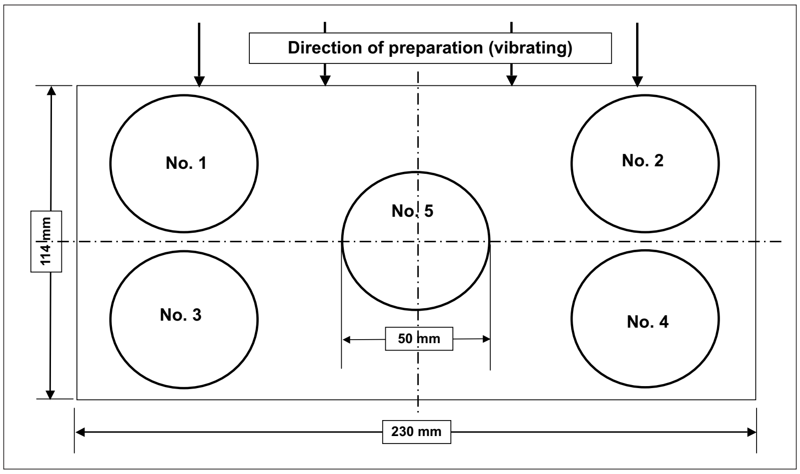

Fig. 3 Scheme of drilling out cylindrical test specimens (H = 50 mm, Ø = 50 mm) from a vibrated test specimen shape A (230 mm x 114 mm x 64 mm)

4 Conclusions

John P. Willi, the retired chairman of ASTM Committee C08 on Refractories, wrote an interesting contribution in ACerS Bulletin [3] regarding personal difficulties in standardization work in the US and internationally. Similar situations exist in Europe too and can be summarized in the following facts:

- Standardising work is often done by retired people. Normally they have a great deal of experience and sufficient time but mostly not enough money for travelling to participate in international meetings at their own expense. Skilled and younger successors are needed urgently.
- Refractory producers are not supportive enough of the development of standards
- financially,
- in terms of the necessary investments for proper equipment in their labs and
- releasing skilled people for such work.
- International standardising work is connected with investigations in local laboratories. The results should be evaluated in open discussions!
- However, international discussions cannot be conducted via e-mail only. This means travelling is a must and boards of producers should spend money to allow their best people to participate in such meetings.

On the other hand, a lot of pre-tests and publications have been made regarding the international standardization of unshaped refractories [4, 5]. In the present paper additional aspects were investigated. Today an enormous technical background is available and final ISO documents could be written tomorrow – if only all countries (and special industries like petrochemical, powergen, etc.) would act in concert! Let's do it! All other arguments and longer waiting for more investigations and results only cost money, time and a lot of unnecessary trouble.

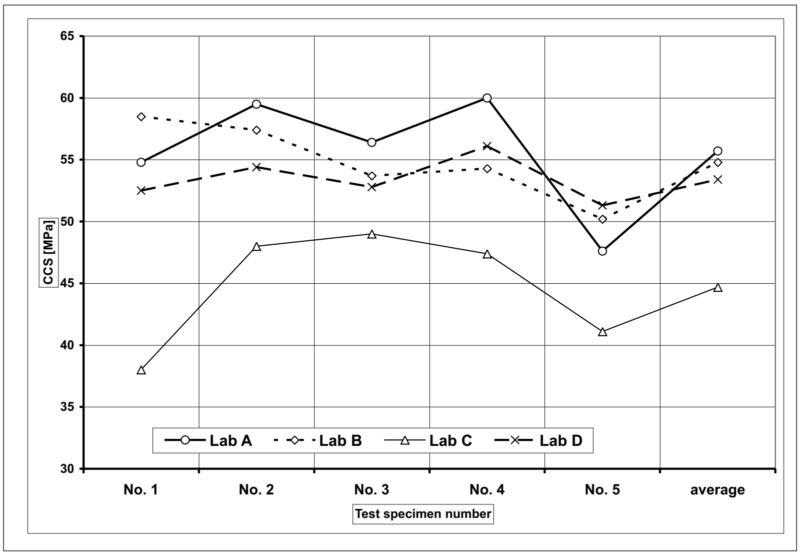

Fig. 4 Comparison of drilled cylinders CCS measured in different laboratories

Tab. 4 Bulk densities of cylindrical test specimens after drying

Test specimen	No. 1	No. 2	No. 3	No. 4	No. 5	Average
Lab A	2,23	2,24	2,21	2,23	2,19	2,22
Lab B	2,24	2,25	2,19	2,22	2,19	2,22
Lab C	2,22	2,23	2,2	2,2	2,18	2,21
Lab D	2,23	2,23	2,19	2,21	2,19	2,21

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