Evaluation of Test Methods for Refractory Bricks: A Comparison and Validity Study of some Cold Crushing Strength Standards

Part 2: Procedure and Determination of Precision

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Following Part 1 of this publication series which dealt with the procedure and the determination of precision of Cold Crushing Strength (CCS) data on several refractory brick grades, part 2 deals with the comparison of ISO 10059-1/EN 993-5 and ASTM C133. This includes the influence of specimen shape (50 mm cylinders and 50 mm cubes) load rate (1,0 MPa/s and 0,2 MPa/s) and a so-called packing a cardboard layer between press plungers and specimen. Furthermore to eliminate the influence of varying brick properties over all samples a so-called twin test (with samples taken as direct neighbours within one brick) was investigated.

The test values according to ASTM results in about 25 % lower values due to the utilisation of the packing which influences the state of stresses and strains in the samples (compressive stresses from the test device are transformed into tensile stresses).

Regarding sample geometry cubic samples lead to about 4 % higher values than cylindrical samples for both standards.

For the determination of the influence of the load rate twin (paired) specimen had to be used to get significant results in a statistical sense. The load rate of 0,2 MPa/s compared to 1,0 MPa/s lowers the CCS value about 7 % for the combination with packing as an average for all brick grades. Without packing the difference is not significant.

A conversion between the test methods ISO 10059-1/EN 993-5 and ASTM C133 is possible and reasonable. The generally large scatter of test results therefore also applies to the regression calculation but in individual cases this can be partially compensated for with more tests. The deviation of the mean value decreases with the factor $\frac{1}{\sqrt{n}}$ (standard error). To consolidate this finding further tests are suggested.

Reasons for the measurements resulting in too low or too high values (Mandel's h "–" values resp. Mandel's h "+" values) are discussed as well as influences on the measured values themselves.

6 Introduction

The background of the paper as part of a series of three publications is given in [1]. Mainly CCS is commonly the straight choice to quickly assess the "quality" of a refractory product. While the measurement seems to be rather simple big variations can be achieved due to differences in the measurement procedure. This paper describes the results of the comparison between standardized test methods ISO 10059-1/EN 993-5 and ASTM C133 which mainly differ regarding load speed (10 MPa/s vs. 0,2 MPa/s) and the introduction of a so-called "packing" a cardboard between the plungers and the specimen. For the measurement itself state-of-the-art testing devices are hydraulic presses equipped with load cells in the axis of the force application

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where the force speed is automatically controlled by proportional regulators.

In short, ISO 10059-1 describes CCS measurement on plan parallel cylinders with a diameter and height of 50 mm and/or cubes of the same length. For shaped bricks the specimens are taken in the same direction of pressing and through the testing machine a load speed of (1,0 ±0,1) MPa/s until break point of the test piece is applied. The measurement is done without use of interlayer packing. EN 993-5 gives information on precision and systematic deviation.

ASTM C-133-97 (2015) (7) describes the procedure of vertical pressing the specimen cubic or cylindric specimens 51 mm (2 inches) in diameter and height. ASTM C133-97 requires the placement between the sample and the plunger of a fibre wall board packing.

The focus of the investigation has been the evaluation of comparison of these two test methods as described in [12]. This included measurements on six brick grades from industrial production (magnesia carbon magnesia spinel bauxite andalusite fireclay and lightweight fireclay) being tested in seven internationally well-respected laboratories.

Tests with ISO cubes ASTM cylinders and ASTM cubes were compared with the reference testing method of ISO cylinders following the methodology described in [12].

The results of the different types of tests have been evaluated in two ways:

- by comparison of the mean CCS values of the bricks and
- by direct comparison of CCS values of so-called twin test specimens (test specimens taken directly next to each other from a brick reducing the influence of brick inhomogeneity and providing more robust results).

The "t-test" was applied to verify statistical equivalence. When statistically there is a significant difference the assessment results as "not equivalent". If statistically there is no difference the assessment results as "equivalent". If statistical there is no certainty the assessment results as "no decision" in which case further investigations (a higher number of test values) would be required to gain certainty.

From the results a linear regression of the differences was calculated for quantification and possibly to allow a conversion between the test methods.

6.1 Test of equivalence

These tests were performed after elimination of outliers by the comparison of the mean CCS values of the bricks using the software prolab+.

6.1.1 Comparison: ISO cylinder to ASTM cylinder

The detailed statistics for the comparison of the ISO cylinder with the ASTM cylinder tests are summarised in Tab. 9.

Since for all grades the t-value exceeds the critical value for the t-test (test decision row) it indicates "not equivalent" results for ASTM cylinder and ISO cylinder. This means that statistical there are differences between measuring CCS on ISO cylinders and on ASTM cylinders.

The difference between the two methods was analysed using the accuracy profile Fig. 21. The curve (blue line) represents the mean difference between ASTM and ISO CCS measurements versus the absolute CCS values obtained. The red lines are the 95 % confidence interval. The variation is

Tab. 9 Detailed statistics and test on equivalence for all grades and ISO cylinder and ASTM cylinder (95 % confidence level)
s.d. = standard deviation

	Grades	A	B	C	F	R	S	Across All Samples
ISOcyl	No. of laboratories	7	7	7	7	7	7	
	Mean [MPa]	89,0	134,0	59,0	72,0	31,8	78,8	
	Reproducibility s.d. [%]	11,7	9,1	9,4	10,5	7,5	13,4	
	Repeatability s.d. [%]	8,3	6,6	7,0	7,2	6,0	10,3	
	Standard error [%]	4,5	3,5	3,6	4,1	2,9	5,2	
ASTMcyl	No. of laboratories	7	7	7	7	7	7	
	Mean [MPa]	70,0	101,7	50,6	59,3	26,9	64,7	
	Reproducibility s.d. [%]	15,2	20,9	12,4	11,6	12,5	15,8	
	Repeatability s.d. [%]	9,5	7,7	7,3	6,0	8,0	11,0	
	Standard error [%]	5,9	8,1	4,8	4,5	4,9	6,1	
Level of significance [%]	5,0	5,0	5,0	5,0	5,0	5,0	5,0	
t-test	t value	3,293	3,401	2,564	3,208	3,079	2,488	6,728
	Critical value	2,179	2,228	2,179	2,179	2,201	2,179	1,96
Test on equivalence	Max tolerated theoretical difference [%]	±10,0	±10,0	±10,0	±10,0	±10,0	±10,0	±10,0
	Max tolerated empirical deviation [%]	±1,4	±1,2	±1,8	±1,8	±2,1	±1,2	±5,7
	Empirical deviation [%]	–21,4	–24,1	–14,1	–17,7	–15,5	–18,0	–18,5
Test decision		not equivalent	not equivalent	not equivalent	not equivalent	not equivalent	not equivalent	not equivalent

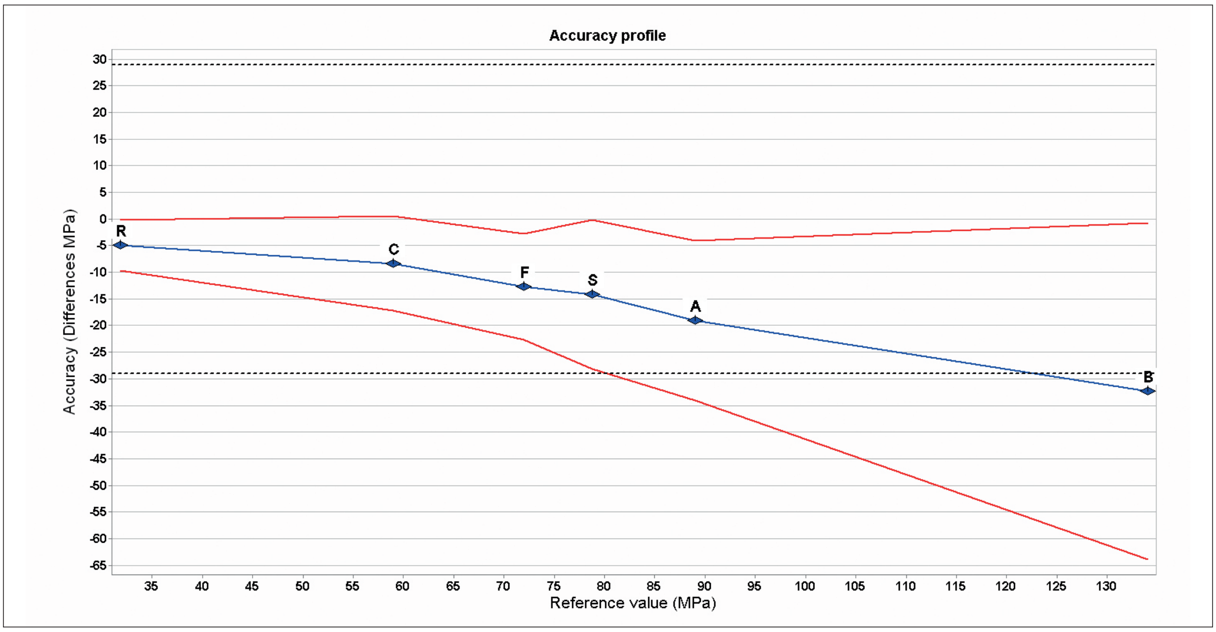

Fig. 21 Accuracy profile: mean values of the absolute differences [MPa] of the test methods ISO cylinder to ASTM cylinder (blue line); the red lines are the corresponding 95 % confidence interval

rather high and the differences are statistically significant. One can expect to get about 25 % lower results if the cylinders are tested according to ASTM. As an example which can be read from Fig. 21 grade A has a mean CCS of 89 MPa and a mean difference between ASTM and ISO of –19 MPa ranging from –4 to –34 MPa, respectively (95 % interval).

6.1.2 More comparisons of cylinders and cubes

The comparison of the CCS measurements on ISO cylinders with the other methods have been carried out with the same t-test approach described in Ch. 2.1.1 for the comparison of ISO cylinders with ASTM cylinders. The result of these comparisons can be summarised as follows:

ISO cylinder to ISO cube	no decision possible
ISO cylinder to ASTM cube	not equivalent in a strict statistical sense
ISO cube to ASTM cube	not equivalent in a strict statistical sense
ASTM cylinder to ASTM cube	no decision possible

The statistics regarding the differences between cylindric and cubic samples for ISO and ASTM did not show clear results indicating too high variations or an insufficient number of results.

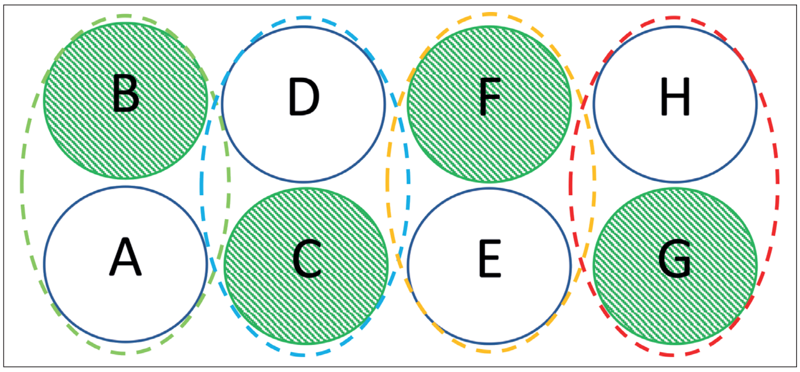

Fig. 22 Sampling scheme of "twin samples" within a brick

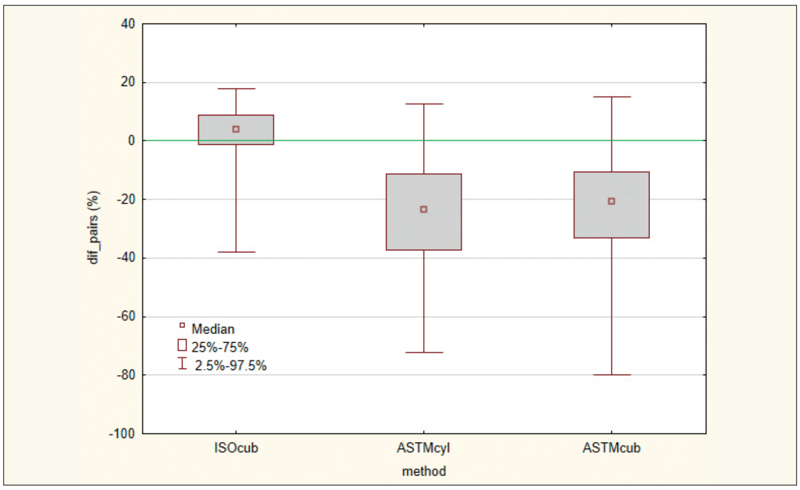

Fig. 23 Box plot of relative differences of ASTM cubes ASTM cylinders and ISO cubes to ISO cylinders with the boxes ranging from 25 % (= Q1 … quartile 1) to 75 % (= Q3 … quartile 3) so called Interquartile Range (IQR) which includes 50 % of all results and the whiskers from 2,5 % to 97,5 % of the results

Tab. 10 Relative differences [%] of median of all test methods to the reference ISO cylinder for each grade separately

	Grades (Difference on Twins)						
Grade	All	A	B	C	F	R	S
ASTM cube [%]	–20,6	–34,8	–26,3	–15,9	–21,7	–9,9	–22,6
ASTM cyl. [%]	–23,4	–36,1	–38,9	–17,4	–23,8	–12,6	–27,2
ISO cube [%]	3,9	8,5	2,9	4,7	5,5	3,5	–1,0

Tab. 11 Relative differences and their deviation of ISO cubes ASTM cubes and cylinders to the referential ISO cylinders

Method	Mean [%]	Quantile 2,5 %	Quantile 97,5 %	Median [%]	Q1 25 %	Q3 75 %	Number of Values
ASTMcub	–22,1	–70,3	14,8	**–20,6**	–32,9	–10,6	226
ASTMcyl	–25,1	–71,8	12,1	**–23,4**	–37,2	–11,4	227
ISOcub	0,5	–37,5	17,3	**3,9**	–1,2	8,7	227

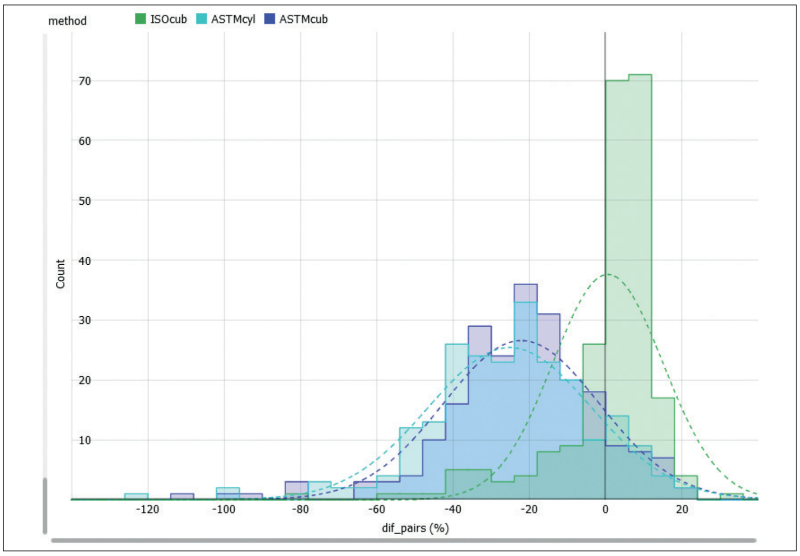

Fig. 24 Relative differences [%] for twin specimen to all other methods included

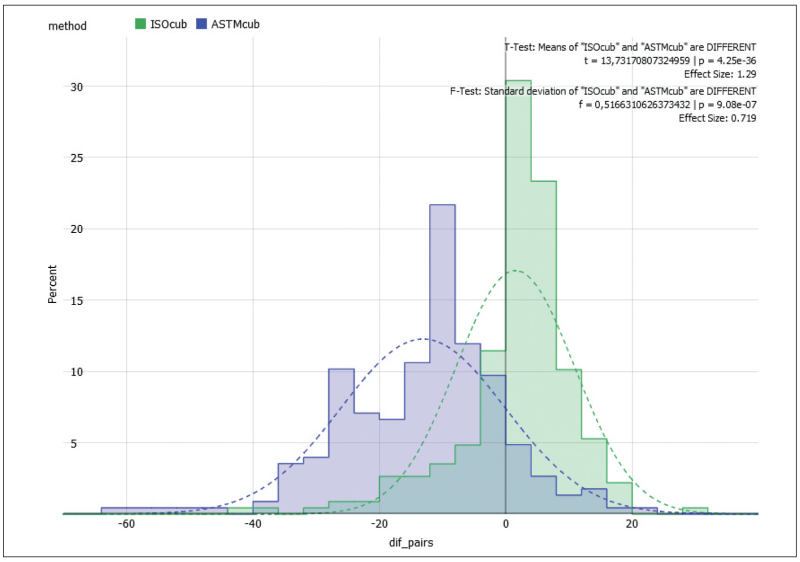

Fig. 25 Comparison of ASTM and ISO on cube specimens and twin samples showing significant different distributions due to mean value and scattering

6.2 Comparison of the methods based on twin samples

In a further step the comparison of the ISO cylinder method to the ISO cube ASTM cylinder and ASTM cube method was evaluated on the differences between the CCS values of twin samples (direct neighbours within one brick, Fig. 22).

The box plot of the relative differences of CCS to the reference (ISO cylinder) indicates that the results of both ASTM geometries are significantly different to that of the ISO cylinders since the 50 % boxes clearly deviate from zero difference line (Fig. 23).

If the CCS test for an ISO cylinder gives about 100 MPa a hypothetical test using an ASTM cylinder would give 25 % of them below 62,8 MPa (Q1 = lower end of the box in Fig. 23) and 50 % below 766 MPa (median = border between gray and black part of the box in Fig. 23).

The deviation was observed for all grades studied Tab. 10. For both standards cubic samples yield slightly higher results i. e. the median with ISO cubes is about 4 % higher compared to the ISO cylinders. However this was stated but cannot be explained well as the difference is not statistically significant.

Values of ASTM cubes are 21 % lower and ASTM cylinders are 23 % lower than ISO cylinders Tab. 10.

The results for the other test methods can be inferred from those of the ISO cylinders by considering the linear regression (Fig. 26). For all brick grades there is a sufficient correlation between ISO cylinder strength and the other strength values. Generally, ASTM test methods give lower values than ISO test methods which can mostly be attributed to the different stress behaviour (Fig. 1) [12].

The deviation for the magnesia spinel brick may have been caused by the influence of the relaxation produced by microcracks (due to thermal expansion mismatch between magnesia and spinel).

Tab. 11 gives the statistic values obtained for all brick grades of the differences for comparison of ISO cylinders with the other shapes investigated.

The comparison shows that when compared to ISO cylinders the highest difference is found for both ASTM methods which also show a higher scattering of their results. This may also be explained by use of the "packing" layer. ISO cubes show similar

results as ISO cylinders (except for magnesia spinel bricks Tab. 10). ASTM cubes and ASTM cylinders also show similar results both well below the values for ISO shapes.
A direct comparison between twin samples gives a distribution of the relative differences over all laboratories and brick grades (Fig. 24).
The other comparison based on cylinders or independent of shapes are only different regarding the mean value not for the scattering of values.

6.3 Simple linear regression between methods on twin specimen

Based on the mean values for each grade obtained from all laboratories a simple linear regression was calculated (Tab. 12).
The difference in results correlates well with the level of the test value. A conversion between the test methods according to the straight line equations of Tab. 12 is possible and reasonable. The generally large scatter of test results therefore also applies to the regression calculation but in individual cases this can be partially compensated for with more tests. The deviation of the mean value decreases with the factor $1/\frac{1}{\sqrt{n}}$ (standard error).
The only exception are the results of the ISO cubes the differences between cube and cylinder are so small that this is completely covered by the measurement uncertainty and thus there is no robust regression.

Tab. 12 **Key figures of the linear regression analysis of ISO cylinders compared to the other methods**

ISO cyl to:	Intercept d	Slope k.x	R²	RMSE
ASTMcube	6,157	–0,318	0,95	1,66
ASTM cyl	8,811	–0,400	0,96	1,76
ISO cube	0,846	0,014	0,03	2,66

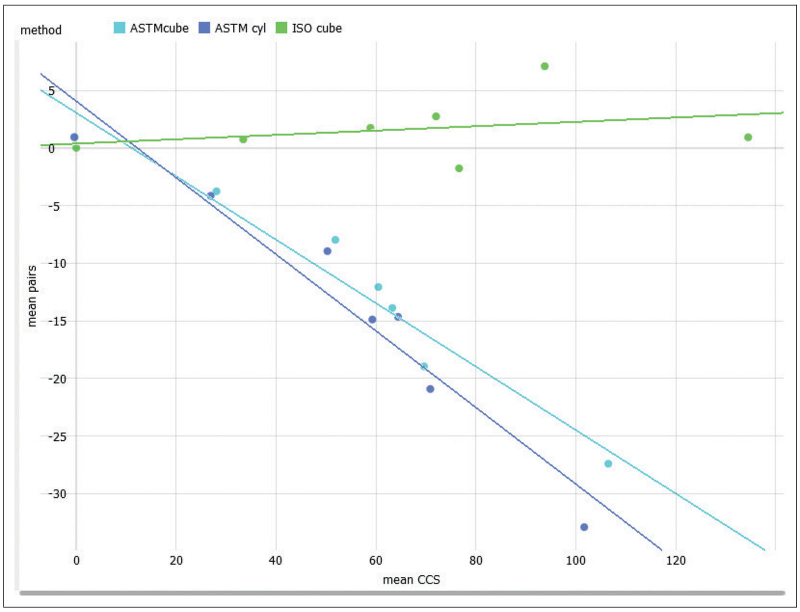

Fig. 26 **Linear regression for the different methods compared on twin specimens for ISO cylinder specimens; one point is the mean value of all results of all laboratories for one grade**

6.4 Comparison of load rate

6.4.1 Comparison of overall measurements

A priori a higher load rate would result in higher strength values and thus this parameter was checked for all brick grades. The evaluation was carried out on cylinder specimens all tests without packing. The load rates used were 1,0 and 0,2 MPa/s, respectively according to the standard specifications Tab. 13. A tendency of lower strength values with a lower load rate could be taken from Tab. 13. Comparative values of the two different load rates of 0,2 MPa/s (ASTM) and 1,0 MPa/s (ISO/EN) based on the shape of cylinders without packing are given in Tab. 13.

Tab. 13 **Comparison of the different load rates based on cylinder tests without packing**

	Grades (Shape: Cylinder; with Packing; Median Bricks)						
Grade	All	A	B	C	F	R	S
0,2 MPa/s [%]	–0,1	–0,5	–3,8	–3,8	–9,9	1,3	3,1
1,0 MPa/s [%]	70,4	90,2	133,0	58,5	70,5	32,0	77,9

Tab. 14 **Comparison of the different load rates based on cylinder with packing**

	Grades (Shape: Cylinder; with Packing; Median Bricks)						
Grade	All	A	B	C	F	R	S
0,2 MPa/s [%]	–4,2	–1,0	–2,9	–1,9	11,4	–3,9	–4,3
1,0 MPa/s [%]	60,5	71,3	105,6	53,1	54,6	29,0	68,0

The highest difference can be observed for fireclay bricks (grade F) where the lower load rate resulted in lower strength values. Contrary magnesia spinel bricks (grade S) show a higher strength on the application of a lower load rate. Thus there is no general statement possible to the influence of the load rate but this must be considered for further action.
The same evaluation based on cylinder shape specimens with packing was carried out to find the effect of the interlayer which influences the state of strain in the specimen (Fig. 1) [12].
Packing reverses the results for fireclay and magnesia spinel bricks probably indicating a special influence by their microstructure. The influence on other brick grades was less pronounced and can be explained by their standard deviation.

6.4.2 Measurement on twin specimen

The evaluation based on twin specimen showed a more precise result when compared to overall measurements. Tab. 15 shows the relative differences of the differ-

Tab. 15 The relative differences of the median values for each grade calculated based on cylinders performed without packing with a load rate of 0,2 MPa/s

	Grades (Shape: Cylinder; with Packing; Difference on Twins)						
Grade	**All**	**A**	**B**	**C**	**F**	**R**	**S**
Cube [%]	–2,3	–5,0	–2,5	0,6	–2,9	–3,3	2,6

Tab. 16 The relative differences of the median values for each grade calculated based on cylinders with packing compared to the method "ISO cylinder" performed without packing. Calculation of the real differences between the load rates –23,39 % and –16,05 % gives 7,34 % (see insert first row)

	Grades (Shape: Cylinder; with Packing; Difference on Twins)						
Grade	**All**	**A**	**B**	**C**	**F**	**R**	**S**
Difference [%]	–7,3	–17,9	–19,0	–5,3	–8,0	–2,7	–8,5
0,2 MPa/s [%]	–23,4	–36,1	–38,9	–17,4	–23,8	–12,6	–27,2
10 MPa/s [%]	–16,1	–18,2	–199	–12,1	–15,8	–9,9	–18,8

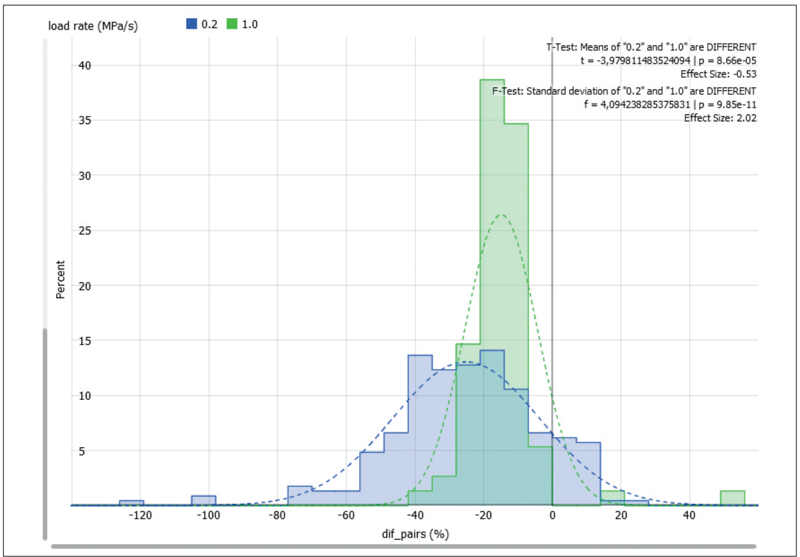

Fig. 27 Distributions of the two different load rates based on cylinders with packing and over all grades giving significant differences of mean values and scatter

ent methods compared to ISO cylinder without packing and at a load rate of 0,2 MPa/s. For measurements on cylinders with a load rate of 0,2 MPa/s and without packing the differences were about –2 % and in a range of +2,6 % and –5,0 % for the grades separately. These values lie within the statistical range and cannot be considered significant.
A different picture results when a packing interlayer on top and bottom of the specimen is used (Tab. 16). The values show that the packing increases the differences.
Considering the case of packing CCS values measured at lower load rates resulted in lower values and the scattering of data was significantly higher. That was verified by the t-test and F-test (Fig. 27). This is in accordance with theory where a lower load rate results in lower CCS values as the stresses and strains have more time to dissipate in the structure with the result of lower strength. Contrarily with a higher load rate the structure is overrun by the stress and cannot react fast enough by destruction thus a higher strength results.
A load rate 0,2 MPa/s with packing has a significant influence of –7,3 % compared to 1,0 MPa/s. This is much more than without packing. Packing dramatically increases the influence of load rate.

6.4.3 ANOVA Analysis on the influence of load rate

As was shown in 6.4.2. only the more precise evaluation method of twin specimens allowed the detection of the significant

Tab. 17 ANOVA for all variated parameter based on all absolute CCS results
Horizontal data (red): Parameter has significant influence on CCS (p-value <0,05)

Effect	**Step of Effect**	**Parameter**	**Standard Error**	**t**	**p**	**–95 % Confidence**	**+95 % Confidence**
Constant		70,1	0,27	259,95	0,00	69,58	70,64
Laboratory (reference 6)	3	1,1	0,58	1,83	0,07	–0,08	2,22
	1	–1,1	0,59	–1,84	0,07	–2,25	0,07
	7	6,5	0,50	13,05	0,00	5,50	7,45
	2	–6,9	0,50	–13,89	0,00	–7,93	–5,97
	4	–3,0	0,58	–5,19	0,00	–4,18	–1,89
	5	–1,1	0,59	–1,78	0,08	–2,21	0,11
Grade (reference R)	S	1,5	0,46	3,23	0,00	0,58	2,37
	C	–16,0	0,43	–37,51	0,00	–16,87	–15,19
	A	11,3	0,56	20,41	0,00	10,26	12,44
	B	51,4	0,56	92,41	0,00	50,28	52,47
	F	–6,2	0,56	–11,13	0,00	–7,28	–5,10
Sample shape	Cylinder	–0,7	0,26	–2,55	0,01	–1,18	–0,15
Load rate	1,0 [MPa/s]	0,1	0,40	0,18	0,86	–0,71	0,85
Packing	no	6,8	0,40	17,18	0,00	6,02	7,57

Tab. 18 ANOVA for all variated parameter based on all differences of twin pairs
Horizontal data (red): Parameter has significant influence on CCS (p-value <0,05)

Effect	Step of Effect	Parameter	Standard Error	t	p	−95 % Confidence	+95 % Confidence
Constant		−8,70	0,52	−16,82	0,00	−9,72	−7,69
Laboratory (reference 6)	3	1,84	1,11	1,65	0,10	−0,35	4,02
	1	0,54	1,13	0,48	0,63	−1,67	2,75
	7	−0,07	1,02	−0,07	0,94	−2,07	1,92
	2	−0,95	1,02	−0,92	0,36	−2,95	1,06
	4	−0,24	1,11	−0,21	0,83	−2,42	1,95
	5	−0,78	1,13	−0,70	0,49	−2,99	1,43
Grade (reference R)	S	0,17	0,90	0,18	0,85	−1,60	1,93
	C	1,25	0,84	1,48	0,14	−0,40	2,90
	A	−1,15	1,12	−1,03	0,30	−3,34	1,04
	B	−1,23	1,12	−1,10	0,27	−3,42	0,96
	F	0,07	1,12	0,07	0,95	−2,12	2,26
Sample shape	Cylinder	2,33	0,50	4,69	0,00	1,36	3,31
Packing	no	11,41	0,75	15,20	0,00	9,94	12,88
Load rate	1,0 MPa/s	3,86	0,75	5,13	0,00	2,39	5,33

influence of load rate for tests performed with packing. ANOVA calculations performed with the software Statistica (TIBCO Software) give a deeper insight into the statistical values. Tab. 17 shows the calculation on absolute CCS for all variated parameters while Tab. 18 shows the same for twin paired measurements.

In Tab. 17 all parameters including grade and laboratory have influence on the absolute values of CCS which may be anticipated. However when the relative differences of twin pairs are calculated only shape packing and load rate are significant (Tab. 18).

Therefore for more precise results a direct comparison of twin specimen verifies shape load rate and packing as significant. Laboratory and grades are not anymore influencing because the database is the relative difference of pairs.

6.5 Comparison of the presence of packing (influence of +lateral confinement)

A first comparison based on median CCS values was done with and without packing and 0,2 MPa/s (ASTM) and 1,0 MPa/s (ISO) (Tab. 19, Fig. 28).

The overall results showed a significant influence of about 15 % of packing. Packing lowers the results between 9–23 % depending on shape grade and load rate respectively.

Tab. 19 Relative differences of the median of bricks with and without packing for both load rates referred to ISO cylinders (ISO standard no packing load rate 1,0 MPa/s) for the different grades as well as averaged over all grades (combined)

		Grades (Shape: Cylinder; Median Brick)						
Packing	**Load Rate**	**All**	**A**	**B**	**C**	**F**	**R**	**S**
without	**0,2 MPa/s [%]**	−0,1	−0,5	−38	−38	−99	13	31
	1,0 MPa/s [%]	70,4	90,2	133,0	58,5	70,5	32,0	77,9
with	**0,2 MPa/s [%]**	−17,6	−21,7	−23,0	−10,9	−13,8	−13,0	−16,6
	1,0 MPa/s [%]	−14,0	−21,0	−20,6	−9,	−22,6	−9,4	−12,8

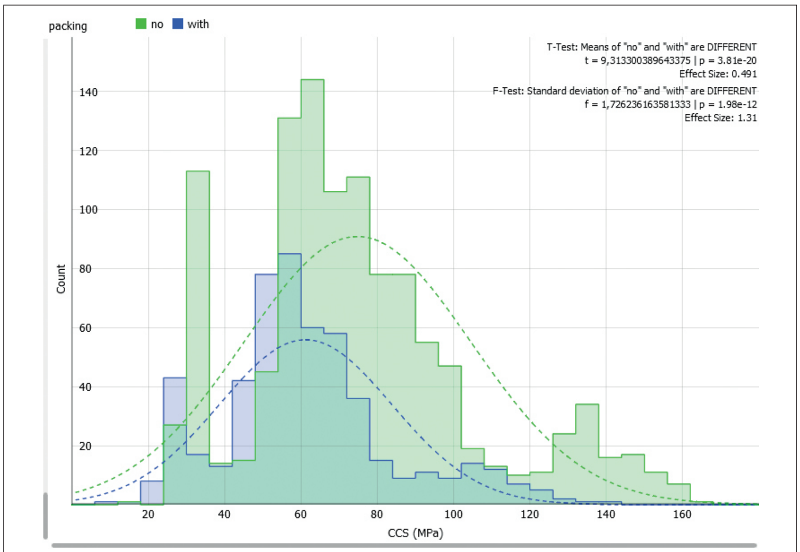

Fig. 28 Histogram for with and without packing overall samples and calculated test for differences of mean value and scatter – both give significant differences

The evaluation without packing at a load rate 1,0 MPa/s of twin cylinder specimens showed a more detailed result especially for the differences between the load rates with packing. These differences were much higher than without packing.

Tab. 20 Relative differences of measurements with packing to ISO (cylinder load rate 1,0 MPa/s without packing) based on twin specimen determination

		Grades (Shape: Cylinder; Difference on Twins)						
Packing	Load Rate	All	A	B	C	F	R	S
without	0,2 MPa/s [%]	–2,3	–5,0	–2,5	0,6	–2,9	–3,3	2,6
with	0,2 MPa/s [%]	–23,4	–36,1	–38,9	–17,4	–23,8	–12,6	–27,2
	1,0 MPa/s [%]	–16,1	–18,2	–19,9	–12,1	–15,8	–9,9	–18,8

Tab. 21 Absolute median values of cylinders tested without packing and load rate 1,0 MPa/s compared to cubes as relative difference of the median tested under the same conditions

	Grades (Shape: Cylinder; Median Bricks)						
Grade	All	A	B	C	F	R	S
Cube dif. [%]	0,5	8,7	3,2	3,4	7,2	4,6	–2,8
Cylinder [MPa]	70,4	90,2	133,0	58,5	70,5	32,0	77,9

Tab. 22 Comparison of cubes to cylinder (load rate 1,0 MPa/s without packing) as relative differences of paired specimen

	Grades (Load Rate 1,0 MPa/s; Difference on Twins)						
	All	A	B	C	F	R	S
Cube [%]	3,9	8,5	2,9	4,7	5,5	3,5	–1,0

The load rate has a high influence when packing is used (–7,3 %) as a comparison of load rate 0,2 MPa/s (–23,4 %) and 1,0 MPa/s (–16,1 %) shows both related to ISO parameters (cylinder load rate 1,0 MPa/s without packing) (Tab. 20). Without packing no significant difference (–2,3 %) was obtained.

This can be explained by the difference in stress modes: with packing the specimen suffers mainly tensile stress (Fig. 1) which will lead to an earlier failure of the sample.

For a load rate of only 0,2 MPa/s the tensile stress has more time to be distributed within the sample which will result in an earlier initiation of fatal cracks.

6.6 Comparison of shapes: cylinder and cube

The two geometries compared were cylinders of 50 mm diameter and 50 mm height and cubes of 50 mm edge length. The initial evaluation was carried out on the results of all bricks.

In Tab. 21, the absolute median values for cylinders tested without packing and a load rate of 1,0 MPa/s are compared to those of cubes tested under the same conditions as relative difference of the median values.

Tab. 22 shows the comparison of cubes to cylinder both tested at a load rate 1,0 MPa/s without packing as relative differences of paired specimen.

Both evaluation methods showed a significant influence of the specimen shape. Cubes yielded higher CCS results of about 4 % differing in the range from –1 % for grade S to +8,5 % for grade A.

These results confirmed those based on simulations found in a master thesis [3].

7 Conclusion

In conclusion more than 1000 single CCS tests have been performed from leading refractory laboratories (producer and independent laboratories) on different refractory grades according to different standards to provide a closer look on this very common test method for refractories. The goal was to statistically compare the results of the different standards (ASTM and ISO) to establish a basis and allow a conversion between the methods and to obtain precision data for all considered test methods. A special drilling schema which ensures a direct comparison of the different methods with samples from one brick (twin or pair specimen) has been applied to avoid possible differences in the CCS of different bricks and to avoid unknown influencing parameters.

The conclusions include the measurements presented in Part 1 of this investigation [12].

7.1 Differences in results between the methods

The main influencing parameters which differ in ASTM and ISO for the CCS test are the load rate and the use of a packing in the ASTM standard. The evaluation shows that the test values according to ASTM results in about 25 % lower values. A more detailed analysis indicate that the reduction is mainly due to the utilisation of the packing which influences the state of stresses and strains in the samples (conversion of compressive stresses into tensile stresses).

The analysis of the sample geometry (cubic vs. cylindrical) has shown that cubic samples lead to about 4 % higher values for both standards.

The load rate of 0,2 MPa/s (ASTM) compared to 1,0 MPa/s (ISO) lowers the CCS value about 7 % for the combination with packing. Without packing the difference is not significant. For this evaluation, it was necessary to switch to twin (paired) specimen to get significant results in a statistic sense or to perform an ANOVA (Analysis of Variance).

7.2 Interlaboratory Study (ILS)

Interlaboratory studies accuracy experiments or round robin tests mean the same and are commonly used to derivate precision data as repeatability and reproducibility for a standard measurement method.

How to organise perform and evaluate an interlaboratory study is very well documented with the standard series of ISO 5725.

Repeatability is comparable over all evaluated test methods (dependency of grades) reproducibility is with ISO and cylinders better than ASTM and cubes.

Even though all laboratories performed tests according to ISO 10059-1 (= EN 993-5:2019-03) and ASTM C133-97 (2015) the statistical error is higher than expected. This was proven by Mandel's h and k statistics [12].

Laboratories 6 and 7 in most cases measure the highest values as can be derived from their Mandel's h values. Mainly measurements according to ISO cylinders and ISO cubes show this behaviour less pronounced are the measurements according to ASTM. Laboratory 2 (and to a lesser extent Laboratory 3) measures in several cases the lowest values and shows a high scattering of results. This is shown by Mandel's k statistics (regarding repeatability) which are not limited to one brick grade and appear very prominent for measurements according to ASTM.

Laboratory 3 shows a similar behaviour but much less pronounced. Still obvious are the extremely high values of measurements for all brick grades on ASTM cubes of Laboratory 3 which cannot be explained by statistical scattering here some systematic error must have occurred.

The determination of repeatability of bauxite bricks (caused by one very low strength value) by Laboratory 5 seems to be an outlier as its results for all other brick grades and parameters are consistent.

The reasons for the observed and detected differences may be manifold and need to be further investigated.

7.2.1 Possibilities the determination of measurements resulting in too high values (Mandel's h "+" values)

Only one reason for the measurement of values being too high is obvious. This is a not centric location of the ancillary adaptor. This can result in the pick-up of forces which results in higher values especially when low forces are present.

Additionally, in case of old measuring devices which measure the force via hydraulic pressure at low forces these are influenced by friction forces which falsify/increase the values. When the former DIN 51067:1977 standard was defined these devices were a standard. The load cells of modern machines measures forces exactly in the direction of the load independently from the oil pressure. Test certificates by external parties also test the devices up to 1 % of the maximum load to proof the compliance of class 1 although only class 2 is required. On renewal of the standard this must be adapted.

7.2.2 Possibilities the determination of measurements resulting in too low values (Mandel's h "–" values)

For the measurement of values being too low several possibilities exist and must be considered permanently by testing laboratories.

Imperfect specimen geometries:

- Uneven face surfaces of the cylinders and cubes resulting in uncontrollable stress peaks
- Not rectangular preparation of the specimen
- Not parallel surfaces of cylinders and cubes
- Pop-outs of the sample surfaces.

Different laboratories also may have more experience in preparation of cylinders compared to cubes so cube measurement results may be more prone to faults.

Influence of steel plates in direct contact with the specimen:

- Worn or nagged and thus uneven steel plates resulting in uncontrollable stress peaks „
- Roughness Ra (0,8–32 µm) can be decreased by permanent measurements
- Hardness due to the lack of the hardness of HRC 58–62 a faster wear may occur.

As a result, a regular revision of the steel plates must be done on permanent use and samples with high strength at least twice a year.

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Remark from the editor:

The last part of the serial including references will be published in refractories WORLDFORUM **16** (2024). For part 1 please see refractories WORLDFORUM **15** (2023) [2] pages 56–67.