Validity Study on Abrasion Resistance Tests Using Calibration to Increase Reproducibility

G.Urbanek, H.-J.Klischat, M. Miranda-Martinez

The Abrasion Resistance Test provides information about a refractory´s resistance to the effect of solid particles impinging on its surface. The ASTM C704/C704M-15 and ISO 16282:2007 standards describe the test of refractories at room temperature. They are similar in type of abrasive grains, specimen size, air flow, and reporting of results. The Task Force "Testing Methods and Standards" of the World Refractory Association (WRA) has investigated abrasion resistance tests by an interlaboratory study, with a focus on test repeatability and precision data. To reduce errors, a calibration procedure was carried out using glass plates. The resulting repeatability interval was r = 22 % and the reproducibility interval was R = 27 %. Confirmed is the quadratic correlationship between abrasion resistance and cold crushing strength. Therefore, as a first approach, cold crushing strength can be used to predict abrasion resistance for the investigated range and grades. This interlaboratory study would strongly recommend to include the precision data obtained from this study into the ISO 16282:2008. Furthermore, it strongly advises the use of a glass plate for calibration and to adjust the air pressure of the equipment to fit the target to 9,3 cm³ ± 0,3 cm³ abrasion of the glass piece.

1 Introduction

The Abrasion Resistance Testing provides information about a refractory´s resistance to solid particles impinging on its surface. Exhaust gases loaded with solid particles are commonplace in the atmospheres of many industrial processes whose vessels are lined with refractories, such as metallurgical furnaces, solid fuel fired power plants, waste incineration plants, petrochemical, cement, and lime industries, etc.

The Abrasion Resistance Test uses as impinging elements silicon carbide (SiC) particles of a defined granulometry. The particles, borne by a defined flow of air, are blasted against the refractory specimen´s surface. The abraded volume, expressed in cm³, is a characteristic value of the material. The higher the abrasion resistance, the lower the abraded volume and vice versa.

Two standards, ASTM C704/C704M-15 and ISO 16282:2007 [1, 2], are currently available for room temperature testing of refractories. They are very similar in type of abrasive grains used, specimen size, air flow, and reporting of results.

Hot Abrasion Resistance testing by ISO 16349 [3]is not part of this investigation.

The Task Force "Testing Methods and Standards" of the World Refractory Association (WRA) has investigated Abrasion Resistance Testing through an interlaboratory study (ILS), which focused on test repeatability and precision data. In this work, more than 168 individual tests, performed by seven renowned laboratories on six different refractory grades were carried. The data obtained were statistically treated, following a methodology that had been previously established by the authors for the analysis of Cold Crushing Strength [4–6] and the improved comparability between measurements of different laboratories [7–8].

In the investigated range a linear correlation was found, and is highlighted, between air pressure and amount of abrasive used. These two parameters can be widely adjusted without significantly decreasing the precision of the method. Air pressure must be carefully checked, since temporary pressure fluctuations, sometimes caused by insufficient air supply, will result not only in poor repeatability, but will also influence test results.

This investigation may lay the ground for a revision of ISO 16282:2007-10, since the standard does not contain any precision data and is very restrictive regarding the

Gerhard Urbanek
RHI Magnesita, Austria

Hans-Jürgen Klischat
Refratechnik Cement, Germany

Manuel Miranda-Martinez
Idonial Technological Center, Spain

World Refractories Association
Brussels, Belgium

Corresponding author: *G. Urbanek*
E-mail: Gerhard.Urbanek@RHIMagnesita.com

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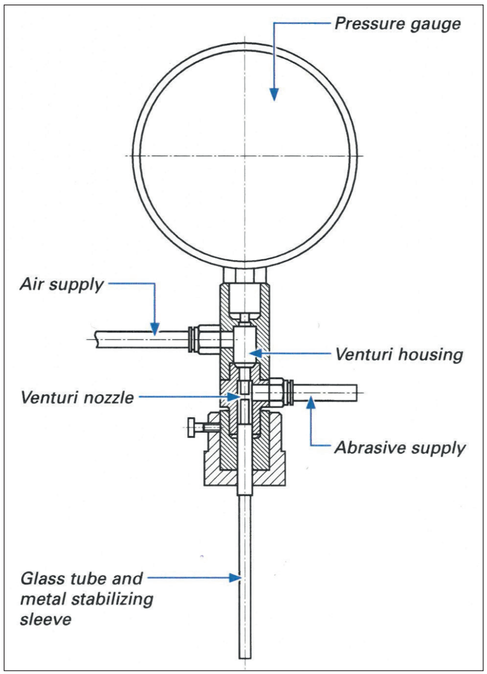

Fig. 1 Abrasion Resistance Test of refractory materials: schematic diagram of the blast gun with venturi system used for measurement (taken from [7])

gunning system. Furthermore, the use of a calibration glass will additionally increase the quality of the results obtained.

2 Description of the interlaboratory study

ISO 16282:2007-10 was followed in the interlaboratory study (ILS) to determine the abrasion resistance. A schematic drawing of the used apparatus is shown in Fig. 1 [7].

As no significant influence was expected from the preparation of test specimens, shapes of 114 mm x 114 mm x 40 mm were cut by each laboratory from the bricks provided. To determine the influence of the surface "firing skin", which is believed to have a beneficial influence on abrasion resistance, firing (external) surfaces and cut surfaces (inner ones) were separately investigated (section 4.2). Fig. 2 shows the locations where the samples were taken from, with outer surfaces A and C corresponding to so-called firing skins and surfaces B and D corresponding to cut surfaces. Each brick gives 8 results, 4 at original and 4 cut surface.

As described in ASTM C704/C704M-15, the abrasion resistance measurement of a glass plate was carried out for calibrating the system. The glass plate abrasion

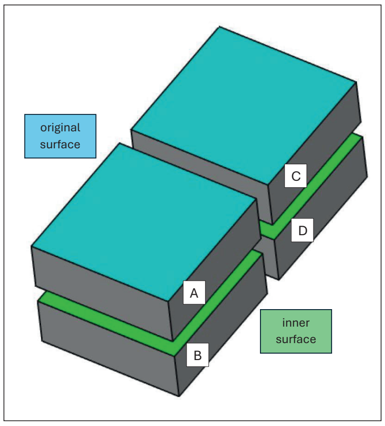

Fig. 2 Schematic drawing showing areas A and C, corresponding to the original (or outer) surface, and areas B and D, corresponding to cut (or inner) surfaces. Each type of surface was subjected to abrasion testing.

resistance was measured before each test series. If the abrasion value of the glass plate was within 9,3 ± 0,3 cm³, 10 samples should be measured before repeating the measurement on a new glass plate. In the case that the value does not fit within the range of 9,3 ± 0,3 cm³, the pressure should be adjusted (usually increased), and another glass plate tested, until the value obtained for the glass plate fits within the range.

3 Laboratories and materials

3.1 Laboratories

Based on their ability to perform testing according to international standards, seven internationally well-respected laboratories were chosen to execute the investigations on the abrasion resistance of refractories. The laboratories were the same as in previous studies regarding CCS testing [4]:

- A laboratory in Germany specialized in measurements on refractory and ceramic materials.
- A laboratory in Poland specialized in measurements on ceramic and building materials.
- A laboratory in Spain specialized in measurements on refractory materials.
- A refractory manufacturer's laboratory in the United States of America.
- A refractory manufacturer's laboratory in Great Britain.
- A refractory manufacturer's laboratory in Austria (which also created the design, performed data preparation and statistical analysis).
- A refractory manufacturer's laboratory in Germany (which also acted as coordinating laboratory).

3.2 Refractory brick grades

Six refractory brick grades were investigated in order to obtain precision data and analyse the comparability of the abrasion resistance measurements. All brick grades were industrially processed into the required shapes using high-performance hydraulic presses. Grades were selected to cover the full range of dense refractory materials and expected material abrasion resistances. Other than the quality of pure lightweight thermally insulating bricks, the grades selected were the same as those whose CCS had been investigated in [4]. Lightweight thermally insulating bricks were excluded from the investigation since they are normally not exposed to abrasive wear (they are used mostly as insulating back-up linings). Still, one lightweight fireclay brick was studied to determine the behaviour in the lower range of abrasion resistance (and low strength resistance).

Tab. 1 Properties of the investigated refractory bricks

Brick grade	Brick C	Brick S	Brick B	Brick A	Brick F	Brick R
Raw material basis	magnesia-carbon	magnesia-spinel	bauxite	andalusite	fireclay	fireclay
Density g/cm³	2,96	3,01	2,88	2,67	2,24	1,78
Porosity %	2,45	14,1	17,50	13,40	16,00	26,70
MgO %	95,50	88,10	0,10	0,10	0,25	0,35
Al_2O_3 %	1,10	10,5	82,80	61,10	41,45	22,30
SiO_2 %	0,80	0,2	10,71	36,30	53,20	68,70
Fe_2O_3 %	0,70	0,50	1,50	0,80	1,40	2,00
RUL: ta/°C		>1700	1500	1700	1400	1350

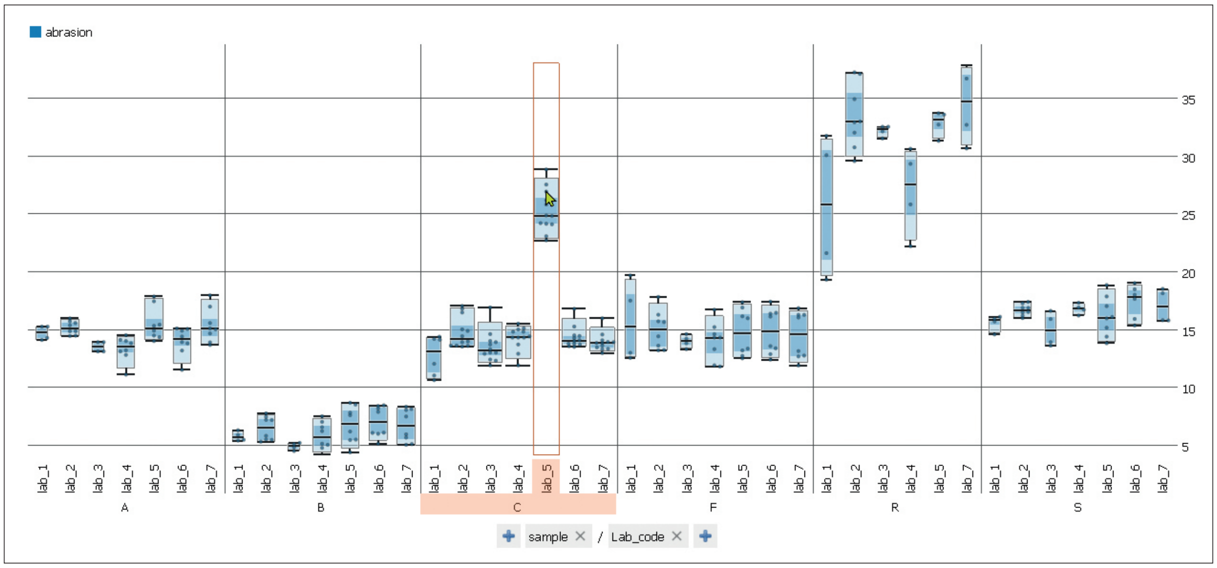

Fig. 3 **Overview of all abrasion resistance test results (cm³). Each box contains 50 % of all results from all laboratories under the defined test conditions, while the whiskers contain 95 % of them**

The brick grades were as follows:

- C – a tempered magnesia-carbon brick containing 10 % graphite,
- S – a dense fired magnesia-spinel brick with an open porosity of 14,5 %,
- B – a fired bauxite brick with 83 % Al_2O_3 content,
- A – a fired andalusite brick with 61 % Al_2O_3 content,
- F – a fired fireclay brick with 40 % Al_2O_3 content, and
- R – a tempered insulating fireclay brick with 22 % Al_2O_3 content.

The main brick characteristics are listed in Tab. 1.

3.3 Test design and sample description

3.3.1 General

To obtain significant and meaningful results, a rather sophisticated procedure, described in section 4, was elaborated to determine possible correlations.

All samples were tested according to ISO 16282:2007-10, while glass plate calibration according to ASTM C704 was applied with additional modification of the air pressure to achieve the desired abrasion [2].

For the bricks of lower abrasion resistance, such as lightweight fireclay bricks (R) and magnesia-spinel bricks (S), only half of the required SiC particles weight was used, and the abrasion resistance value thus obtained was multiplied by 2. The result is the same as with the originally established amount of SiC [7], while the interactions with rebounded abrading particles are reduced. Furthermore, the thickness of the samples could be even smaller without influencing the results.

4 Evaluation of the interlaboratory study

4.1 Overview of all results

Fig. 3 shows the results for all brick grades and laboratories as box plots.

The results show a good general conformity. Bigger differences were observed for the insulating fireclay brick grade R, which, as expected, showed the lowest abrasion resistance. For the magnesia-graphite brick, grade C, a consistent outlier was observed for laboratory 5. Note that not all laboratories performed testing of the two types of surfaces: inner and outer.

4.2 Comparison of the specimens´ surface: outer (fired) surface and inner (cut) surface

The mean value of the results of the original outer surfaces (denominated "out") of all laboratories (first column in Figs. 4 and 5)

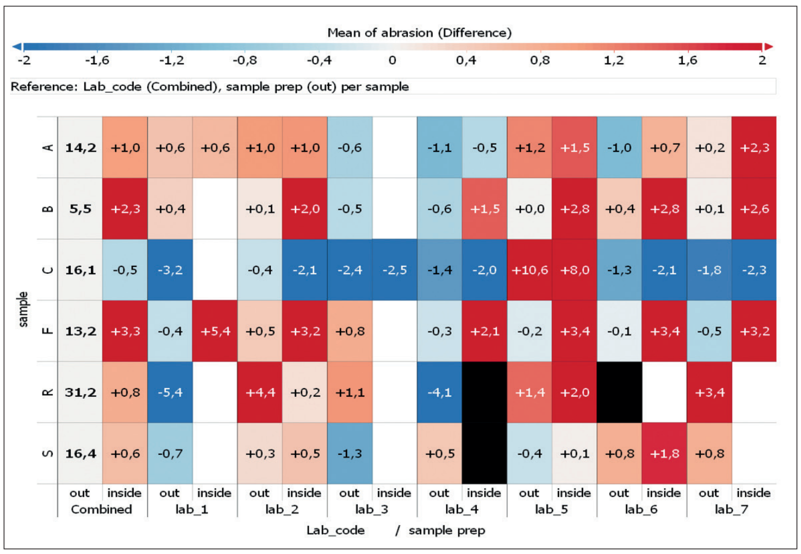

Fig. 4 **Absolute differences in abrasion (values in cm3) for all brick grades and all laboratories, comparing outer (original) and inner (cut) surfaces values to the mean value for the outer surface from all laboratories.**

has been the reference abrasion value for each brick g rade; Fig. 4 shows the absolute differences, and Fig. 5 shows relative differences to this reference.

Despite the fact that not all laboratories measured the inner and outer surfaces, overall, the original surface of fired bricks had a significant higher abrasion resistance than the cut (inner) one, although the differences were not huge.

Looking at the relative values in Fig. 5 these findings are confirmed. As for the relative values, even the deviant measurements of Lab 5 on magnesia-graphite bricks are not striking, as the statistical spread is still low despite the deviation of the mean value.

This can also be derived from Tab. 2: for all fired products (andalusite (A), bauxite (B), fireclay (F), magnesia-spinel (S) brick grades) the outer surface (so-called firing skin) shows a higher (read better) abrasion resistance compared to the cut surface.

The explanation may be that due to the high temperature during firing, a slightly higher degree of sintering may take place on the refractory brick surface, which would provoke a "stronger barrier" against abrasion. This barrier must be removed before further "ablation" takes over at the intrinsic rate of the inner structure.

For unburnt magnesia carbon brick (C) the abrasion resistance of the cut surface was 3 % higher than the original (external) surface. However, the other unburnt brick, insulating fireclay (R), did not show the effect of higher abrasion at the inner surface, but for both brick grades differences between the two different surfaces were rather small and may lie within the experimental error. Some deviations, such as 42,9 % for the bauxite brick, were quite remarkable but in that case the abrasion values were compa-

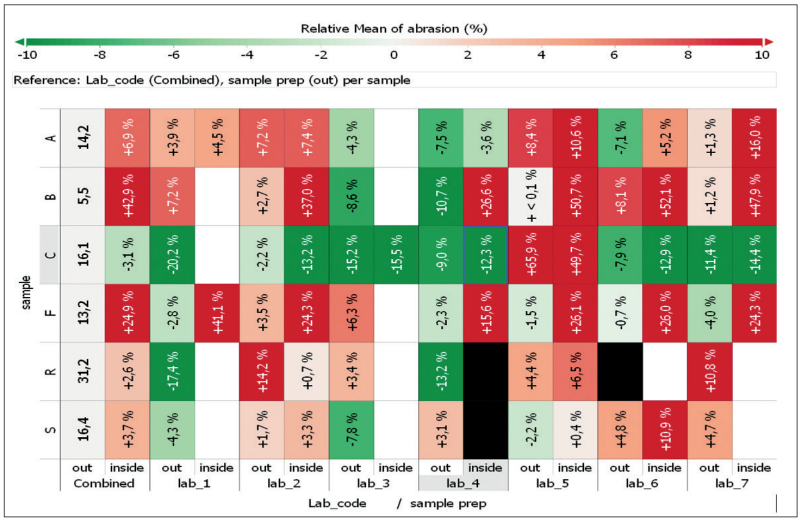

Fig. 5 **Relative differences in abrasion for all brick grades and all laboratories (values in % of mean values of all laboratories – combined columns), comparing the outer and inner (cut) surfaces values to the mean value of the result of the outer surface from all laboratories**

Tab. 2 **Test of equivalence for the abrasion resistance measurements on the outer (original) and inner (cut) surfaces**

		A	B	C	F	R	S	Across all samples
original surface	No. of laboratories	7	7	7	7	6	7	
	Mean	14,25 cm³	5,47 cm³	14,30 cm³	13,20 cm³	31,39 cm³	16,37 cm³	
	Reproducibility s.d.	8,4 %	11,8 %	12,6 %	5,3 %	10,8 %	7,5 %	
	Repeatability s.d.	7,8 %	8,7 %	9,0 %	4,0 %	8,2 %	7,1 %	
	Standard error	3,3 %	4,6 %	4,9 %	2,1 %	4,5 %	2,9 %	
cut surface	No. of laboratories	6	5	6	6	2	3	
	Mean	15,170 cm³	7,815 cm³	13,865 cm³	16,653 cm³	32,314 cm³	17,175 cm³	
	Reproducibility s.d.	6,3 %	9,8 %	10,2 %	6,8 %	4,4 %	10,1 %	
	Repeatability s.d.	6,3 %	6,3 %	4,3 %	5,6 %	3,1 %	5,1 %	
	Standard error	2,7 %	4,5 %	4,3 %	2,9 %	3,2 %	6,0 %	
Level of significance		5,0 %	5,0 %	5,0 %	5,0 %	5,0 %	5,0 %	5,0 %
t-test	t value	1,50	5,44	0,48	6,28	0,52	0,70	5,50
	Critical value	2,20	2,31	2,20	2,31	2,57	3,18	1,96
Test on equivalence	Maximal tolerated theoretical difference	+/- 15,0 %						+/- 15,0 %
	Maximal tolerated empirical deviation	+/- 7,3 %	+/- 3,0 %	+/- 4,5 %	+/- 7,5 %	+/- 5,3 %	+/- 3,9 %	+/- 9,6 %
	Empirical deviation	6,5 %	42,9 %	-3,0 %	26,1 %	2,9 %	4,9 %	18,1 %
Test decision		equivalent in the strict sense	not equivalent	equivalent in the strict sense	not equivalent	equivalent in the strict sense [*]	no decision possible [*]	not equivalent

rably small, thus any difference produce a high deviation.

The fireclay brick showed comparatively higher differences between inner and outer surface abrasion, indicating the formation of a thicker barrier of "firing skin" on the surface due to the higher amount of amorphous phase formed.

Since abrasion values for outer and inner surfaces are different, it should be agreed, or arbitrated for official use, which surface is to be measured and this circumstance must also be mentioned in the documentation.

For determination of equivalence, the statistical "t-test" was applied, Tab. 2. In this case if there is a statistically significant difference between two results, the assessment is "not equivalent". If statistically there is no difference, the assessment is "equivalent". In the case that there is no final statistical certainty, the assessment is "no decision possible" and further investigations (higher number of test values) could provide certainty. A significance level of 5 % was chosen as it is good practice for statistical tests on refractories.

For andalusite, magnesia-carbon, and lightweight fireclay bricks, there is equivalence between outer and inner surface abrasion resistance.

For bauxite and fireclay bricks, there is no equivalence between outer and inner surface abrasion resistance. There are several explanations for it. All are related to each brick´s microstructure: fireclay and bauxite bricks contain varying amounts of amorphous phases, which may contribute to the observed behaviour. Another reason may be related to the position of the brick in the kiln during firing (such as being placed in a zone of shadow on the kiln car stack during firing), the influence of pushing rate, etc. The formation of amorphous phases is further accelerated by the presence of secondary phases in brick grades containing iron oxides, titania, alkali oxides and earth alkali oxides. The behaviour of andalusite bricks is different, since firing temperature is below the transformation of andalusite into mullite and cristobalite, a homogeneous mineralogical structure is present throughout the brick.

A decision of equivalence cannot be made for the magnesia-spinel brick. In this case further tests should be performed to confirm equivalence. Still, these bricks tend to react like other low-amorphous content grades, and their behaviour may also be influenced by the microcrack system created by the thermal expansion mismatch between magnesia and spinel grains [6].

4.3 Final ILS evaluation

Finally, a statistical evaluation was performed to determine the reproducibility and repeatability of the abrasion resistance tests. The aim of the investigation was gathering of statistical data on abrasion resistance rather than an exhaustive assessment of material specific properties.

4.3.1 Evaluation software

The evaluation software used for all tests was PROLab Plus (supplied by QuoData, Dresden/DE). This software was used for planning, organising, performing, and analysing the interlaboratory studies in accordance with DIN ISO 5725-2:2019-12 [9].

4.3.2 Outlier identification and treatment

For statistical evaluation (Tab. 3), the outliers can be easily identified graphically in Mandel's statistics graphs as red bars (section 4.5). Furthermore, outlier tests according to Grubbs and Cochran were carried out according to ISO 5725-2:2019-12, [9]. The Cochran test is applied for the analysis of variance and the Grubbs test is applied for the analysis of mean value deviations.

A distinction in the outlier test is drawn between outliers (at the significance level 1 %) and stragglers (at the significance level 5 %). As a result, the corresponding measured values (only the outliers, not the stragglers) were excluded from further calculations. This includes mainly the outliers for magnesia-graphite bricks from laboratory 5, fireclay bricks from laboratory 4 and lightweight fireclay bricks from laboratory 1.

4.4 Method characteristics

For the evaluation, the standard deviation, reproducibility R, and repeatability r were determined in absolute and relative values for the participating laboratories.

Tab. 4 shows the precision data based on Round Robin Tests among 8 laboratories performed for ASTM. A comparison of Tabs. 3 and 4 clearly shows that the reproducibility interval and, above all, the comparison interval in Tab. 3 are significantly better and narrower. The major difference in the test procedure was that in the current round robin test (Tab. 3) the glass plates were calibrated before the tests, i. e. adjusted with the applied pressure of pressed air. The problem of the differences between repeatability and reproducibility can be seen most clearly in Tab. 4 for the glass plate, where the reproducibility is 4,5 times higher than the repeatability interval. Provided, of course, that "identical" glass plates were used. These laboratory differences were calibrated away in Tab. 3.

Tab. 3 **Method characteristics and test as well as statistical results for abrasion resistance of various refractory brick grades**

Material	Mean value [cm³]	Standard deviation within laboratories [S_r]	Standard deviation between laboratories [s_R]	Repeatability interval [r]	Reproducibility interval [R]	Relative repeatability [%r]	Relative reproducibility [%R]
A	14,2	1,04	1,32	2,92	3,68	20,6	25,9
B	5,5	0,45	0,56	1,27	1,56	23,2	28,5
C	14,3	1,18	1,47	3,30	4,1	23,1	28,7
F	13,2	0,66	0,76	1,86	2,13	14	16,1
R	31,2	3,59	5,17	10,04	14,49	32,2	46,4
S	16,4	1,25	1,35	3,51	3,77	21,4	23
					Median [%]	**22,25**	**27,2**

Tab. 4 Precision statistics for abrasion resistance from ASTM C704/C704M-15 (2)

Material	Mean value [g/cm³]	Standard deviation within Laboratories [s_r]	Standard deviation between Laboratories [s_R]	Repeatability Interval [r]	Reproducibility Interval [R]	Relative Repeatability [%r]	Relative Reproducibility [%R]
High-alumina brick	4,19	0,39	0,71	1,08	1,98	25,80	47,19
Silica brick	22,17	2,64	4,62	7,40	12,95	33,36	58,41
Abrasion-resistand castable	8,36	0,87	1,89	2,42	5,29	28,99	63,24
Super-duty firebrick	25,48	4,25	7,81	11,90	21,86	46,70	85,80
Conventional high-cement castable	10,89	2,12	3,02	5,94	8,45	54,54	77,59
Plate glass standard	9,28	0,34	1,51	0,95	4,23	10,24	45,58

Two single results under repeatability or reproducibility conditions should not differ more than the repeatability or reproducibility interval, respectively; otherwise, it is not the same sample, or the testing methods are different.

Generally, the values for repeatability (within one laboratory only) are lower than for reproducibility (for all laboratories), which can be explained by the presence of more influencing factors for the reproducibility test: operator, equipment, item, method.

For destructive tests, natural material inhomogeneities – such as grain sizes and distribution, agglomerations, pores and their sizes – are directly related to the spread of the results – for s_r within one brick and for s_R between bricks. This circumstance is related to the ILS design.

In this study for fireclay bricks (F) both values, R and r, are lower than those in (7), possibly due to the requirement, for all participating laboratories, to adjust air pressure by prior calibration of test conditions using a glass plate.

The comparably high values of scatter for the repeatability and reproducibility of fireclay brick types are remarkable (as already pointed in Fig. 3). One cause could be a varying amount of glass phase in the refractory structure which could be eroded unevenly, loosening for instance different quantities of coarse grains; a circumstance that would significantly influence the abrasion resistance obtained.

Due to these tighter R and r values (compared to [7]), abrasion resistance test can produce reliable, repeatable and consistent results. The only exception was the outlier of laboratory 5 for magnesia-graphite bricks, but this was eliminated from the reproducibility data (as the repeatability data showed a low statistical spread of results).

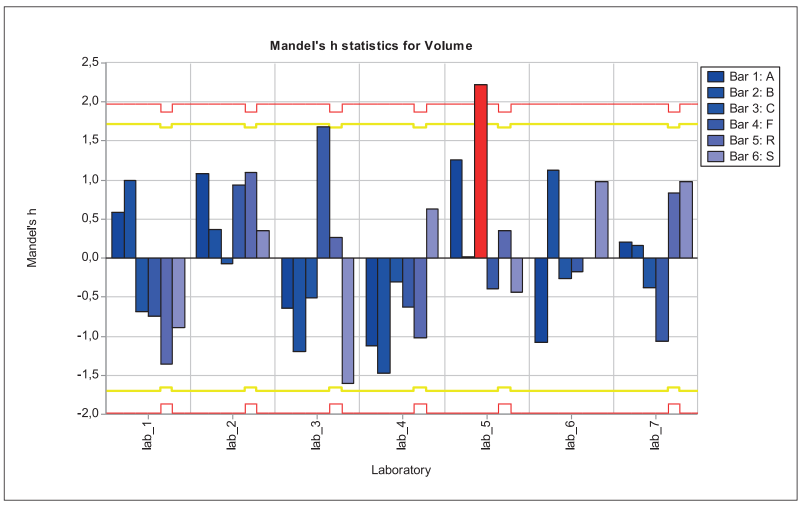

Fig. 6 Mandel's h statistics for abrasion resistance measurements

4.5 Mandel's Statistics

Mandel's k and h test statistics are measures for data consistency, particularly unseful for interlaboratory studies. Differences between samples obtained from different laboratories are used to find outliers of a sample compared to others. As recommended by ISO 5725-2 (9) and ASTM E691 (10), Mandel´s h statistics was used for comparison of the mean values of all laboratories and grades, while Mandel´s k statistics was used for the repeatability performance of each laboratory. These Mandel's statistics for abrasion resistance testing are shown in Figs. 6 (Mandel's h) and 7 (Mandel's k). Bars in yellow and red indicate significant and high variations, respectively.

The h statistics show that all values are within the typical range, except for the already mentioned results from laboratory 5 for magnesia-graphite bricks. Further systematic deviations of laboratories, or bricks, is not obvious.

If a bar appears in yellow or red in a Mandel's k statistics graph (Fig. 7) the repeatability is significantly different from the other laboratories. If it is in red, it is defined as an outlier and is excluded from further calculations of the precision data. In this case, laboratories 1 and 4 showed outliers above the critical value, but not for similar brick grades (laboratory 1 for insulating fireclay brick R; laboratory 4 for fireclay brick F; laboratory 5 for bauxite brick B). All other grades showed no peculiarities regarding the repeatability performance of each laboratory. It is worth noting that the outlier for magnesia-graphite bricks from lab 5 does not show up in Mandel's k statistics, as the values within the laboratory tests show only a normal statistical spread.

4.6 Summary of results for each brick grade

For each brick grade and laboratory, the results are shown in Figs. 8 to 13.

Fig. 8 shows only results for the andalusite bricks. All data are within the tolerance

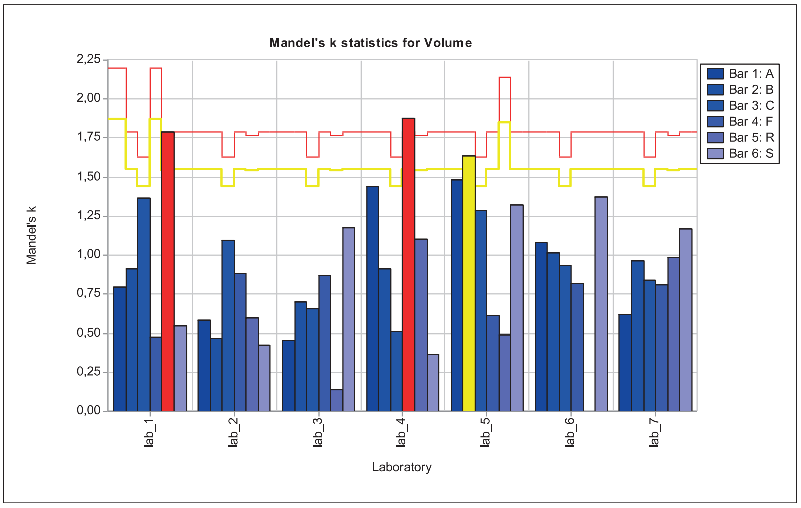

Fig. 7 Mandel's k statistics for abrasion resistance measurements

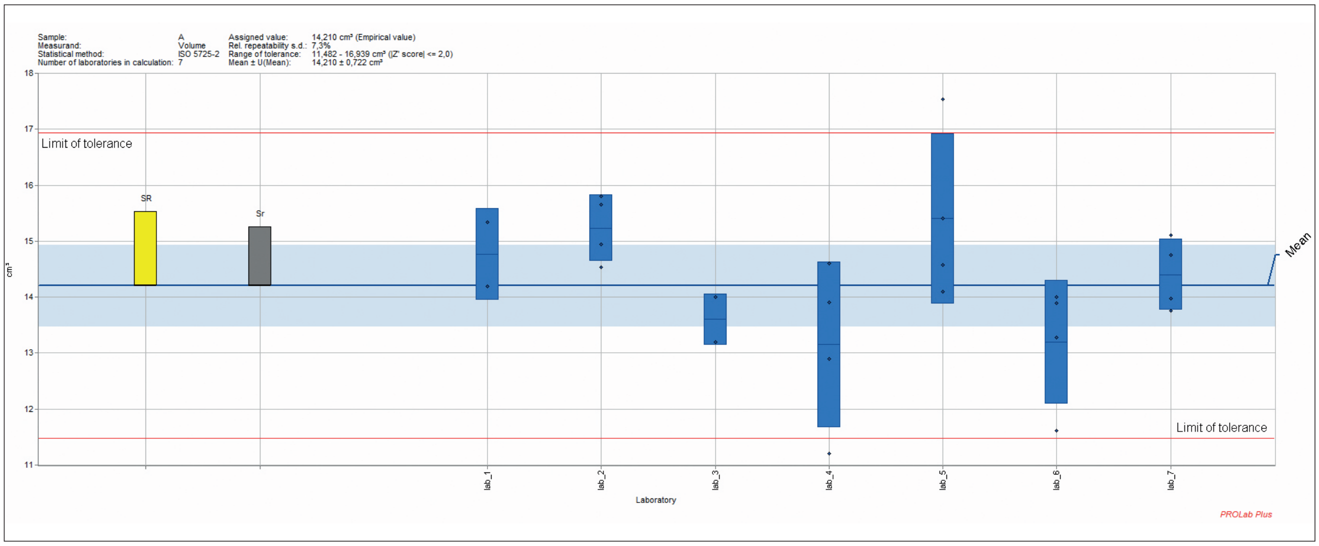

Fig. 8 Detailed graph for the Andalusite brick (A) showing all single results of each laboratory (and grouped by laboratory)

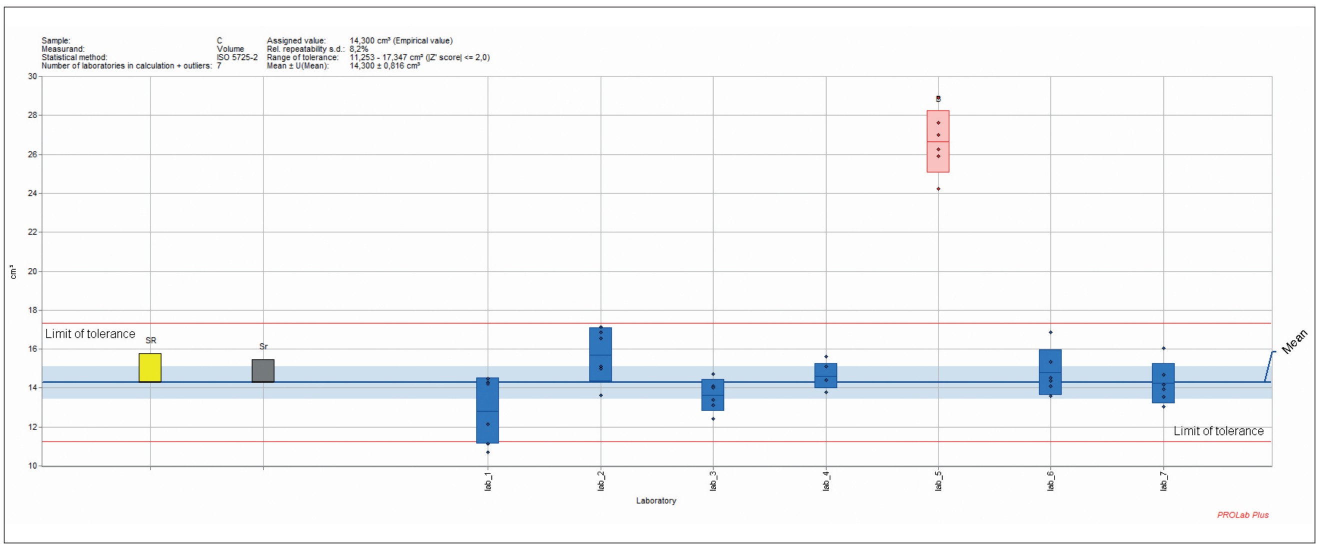

Fig. 9 Detailed graph for the Magnesia-Carbon brick (C) showing all single results of each laboratory (and grouped by laboratory)

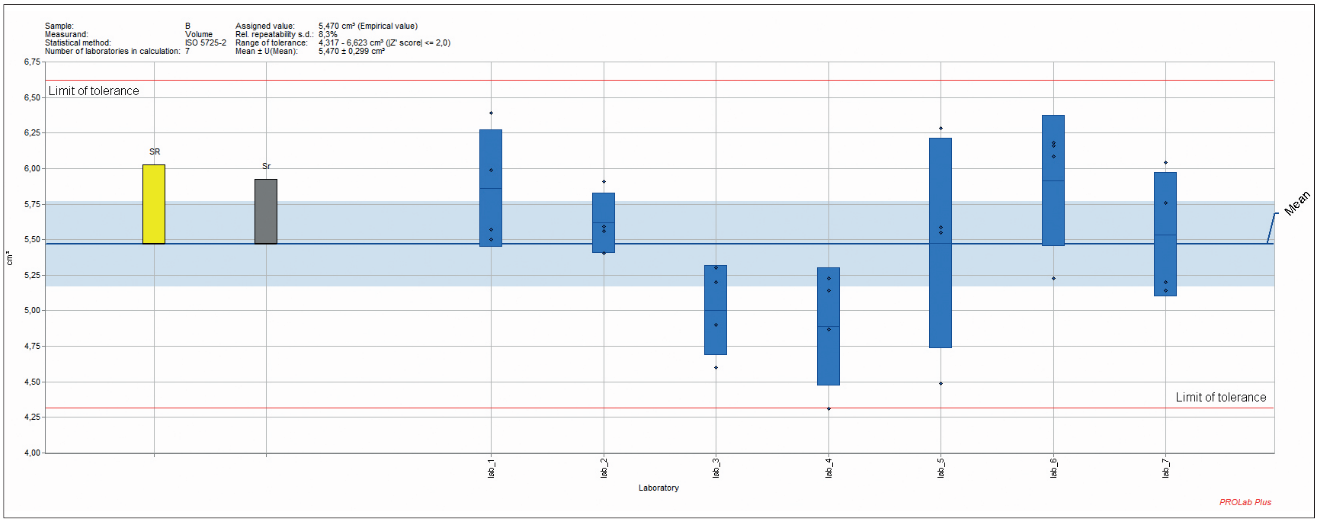

Fig. 10 **Detailed graph for the Bauxite brick (B) showing all single results of each laboratory (and grouped by laboratory)**

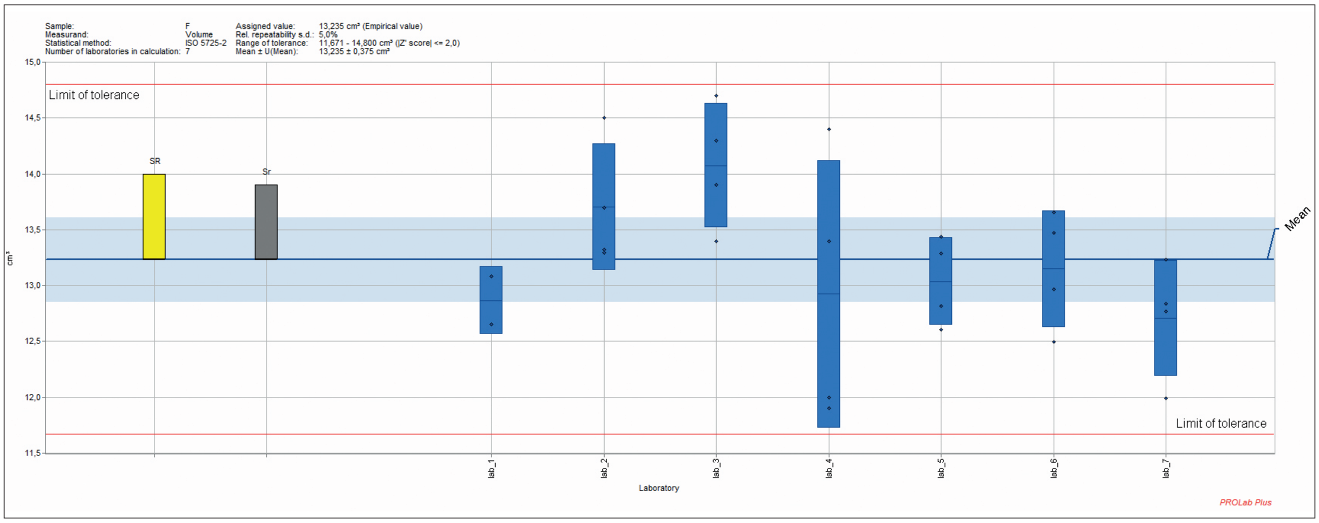

Fig. 11 **Detailed graph for the Fireclay brick (F) showing all single results of each laboratory (and grouped by laboratory)**

limit. A wider spread (>2 cm^3) can be observed for labs 4, 5, and 6. Also clear are the spreads for labs 4 and 5, which differs within certain limits at the lower and upper tolerance limits with their extreme values, which also results in a difference of the median value of approx. 1 cm^3. From these data, no material specific dependencies can be derived.

Fig. 9 shows the results for magnesia-carbon bricks. All data except laboratory 5 are within the tolerance limit, with only a spread of values higher than 2 cm^3 for laboratories 1 and 2. Obvious outliers are the values of lab 5, which not only show a much higher abrasion but also higher spread than the other laboratories. From these data, no material specific dependencies can be derived.

Fig. 10 shows the results for bauxite bricks. All data are within the tolerance limits. Still, the maximum spread of laboratory 5 is 1,5 cm^3, all others are lower than or equal to 0,8 cm^3. Generally, all data are inconspicuous and show no peculiar trend. Also in this case, no material specific dependencies can be derived.

Fig. 11 shows the results for fireclay bricks. All data are within the tolerance limit and the results are as expected. However, Laboratory 4 shows a remarkable spread of values, exceeding 2,5 cm^3. All other laboratories are well grouped around the mean value. The data are inconspicuous and show no peculiar material dependent behaviour, probably the phases forming the material (amorphous phase, cristobalite, and mullite) are distributed evenly within the microstructure and therefore there is no evidence of inhomogeneous behaviour.

Fig. 12 shows the results for the insulating fireclay bricks. Lower abrasion resistance was expected and confirmed by the results. Only laboratory 1 slightly exceeds the tolerance limits, possibly due to the higher abrasion loss for this brick type. The unburnt state of this brick contributes to the comparatively high abrasion observed. A less tightly bonded structure resulting from the use of a phosphatic bond, instead of the ceramic one of the other investigated brick grades, could contribute to a higher abrasion by the hollowing of some big grains. However, except for Laboratory 1, the spread of values is well within the tolerance limit. Still, it is obvious that Laboratory 1 and Laboratory 4 measured values below the mean,

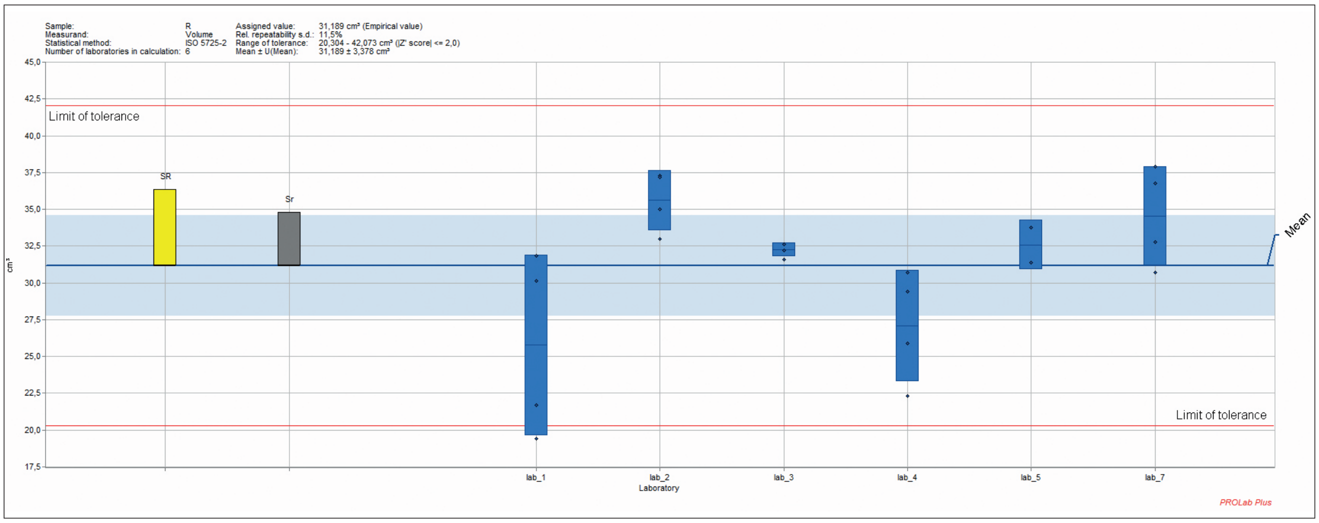

Fig. 12 Detailed graph for the Insulating Fireclay brick (R) showing all single results of each laboratory (and grouped by laboratory)

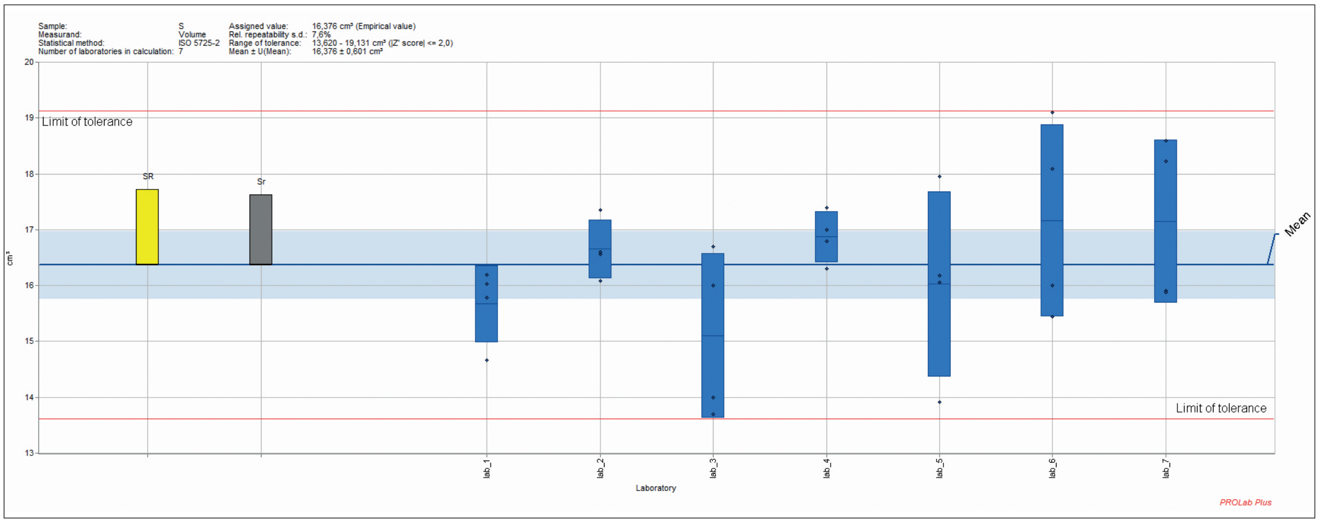

Fig. 13 Detailed graph for the Magnesia-Spinel brick (S) with all single results of each laboratory (and grouped by laboratory)

while all other laboratories measuring values above the mean. Generally speaking, the data are inconspicuous and showed no peculiar behaviour. As material specific dependencies we could mention the softer bond of the grains in their unburnt structures.

Fig. 13 shows the results for the magnesia-spinel bricks, where a special behaviour could be expected due to the designed presence of a microcrack system intended to reduce the Young's modulus, [6]. Nevertheless, the tolerance bars are not wider, indicating a similar standard deviation, and no microstructural influence. Four laboratories showed a deviation around 2 cm³, as was the case for fireclay bricks F. An influence of the microcrack system expected from the thermal expansion mismatch of the brick components was not found in our investigation.

5 Correlation between abrasion resistance and cold crushing strength

Since cold crushing strength is thought to be a property connected to abrasion resistance [11], the behaviour of both properties was correlated in Fig. 14 for the brick grades investigated. CCS data were taken from reference [6] and in this correlation only CCS measurements performed on cylinder samples have been considered [12, 13].

As expected, higher CCS values resulted in higher abrasion resistance. As shown in the case of strength values ranging from 60 and 85 MPa, this can somewhat be nuanced by microstructural particularities, such as type of bonding, grain sizes, etc., which will affect each material´s precise behaviour.

The correlation seems to follow a quadratic function, which is more likely (regression constant R^2=0,88) than a simple linear regression (R^2=0.77). The correlation can be found for CCS measurements on ISO and ASTM cylinders, Fig. 14. This non-linear quadratic function agrees with the results presented in [13] and indicates the finding that a rapid increase in abrasion occurs when a threshold value for strength is underrun [11].

If statements on abrasion resistance of a refractory are needed, it can be confirmed that a first approach could be the consideration of the cold crushing strength. Direct

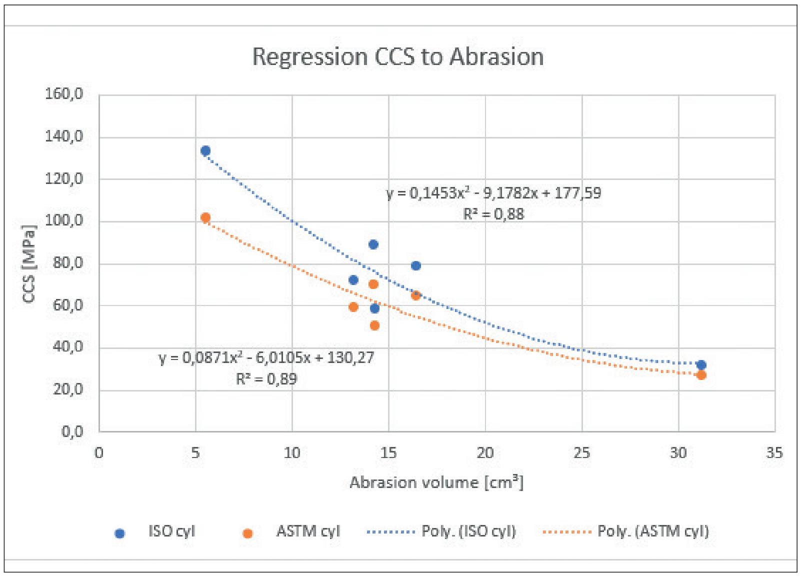

Fig. 14 Correlation of abrasion resistance to cold crushing strength for ISO 10059-1:1992-02 and ASTM C133-97 measurements on cylinders

calculation of abrasion resistance from CCS data still might be defective and should only be done when all possible sources of error are examined.

6 Conclusion

The abrasion resistance of various basic and non-basic refractories, as well as fired and tempered brick grades has been investigated in an Interlaboratory study (ILS). Abrasion resistance tests were performed according to ISO 16282:2007-10 [1]. To reduce errors a calibration procedure was carried out using glass plates. As a result, the repeatability interval obtained was r = 22 % and the reproducibility interval R = 27 %. This was a good outcome, especially for R, and was made possible by using the calibration with glass plates. These values of repeatability and reproducibility are well below those of [2]. Therefore, for consistent measurement of abrasion resistance, glass plate calibration seems to be mandatory to obtain reliable and comparable results.

Despite the positive outcome of the repeatability and the reproducibility intervals obtained, some observations may need further attention in future (Figs. 8–13). Laboratory 1 always measured values below the median, except for bauxite and andalusite bricks where only values above the median value were obtained. Laboratory 2 measured values above the mean value for all brick grades. Laboratory 5 measured values around the mean value for all brick grades, except a strange outlier for magnesia-carbon bricks. All other laboratories did not show any systematic bias. Although all laboratories measured abrasion resistance according to ISO 16282:2007-10 resp. ASTM C704, a supervised interlaboratory study comprising the same laboratories in cooperation with other ISO members could lead to further explanation of the results.

The original surface of the fired samples gave "better" (lower) abrasion results than the cut faces. This is due to the presence of a "firing skin" which protects the surface against wear, at least during a short period of time. Once this "firing skin" is removed, abrasion will go on at a slightly higher rate. That rate will be like that observed for cut surfaces. For unfired fireclay and magnesia-graphite brick grades, the abrasion on the outer surface was slightly higher than that recorded for the cut surface, or at least within experimental error. However, the effect was not very pronounced, and further investigations of this effect might mainly be interesting for scientific purposes. Nevertheless, in case of discrepancies when determining the performance of industrial materials, one should keep in mind this circumstance.

A general explanation for the observed material-specific abrasion values is not obvious. The well-known high abrasion resistance of bauxite bricks (assigned to the presence of phases with high hardness, such as corundum), as well as the low resistance of lightweight fireclay bricks (due to their high porosity (Tab. 1) were confirmed. Also, unsurprisingly, the abrasion values of other bricks grades were intermediate and can be explained by their microstructural characteristics, such as presence of amorphous phases, microcracks, influence of graphite for magnesia-carbon bricks, etc.

Already known, and here confirmed, is the quadratic correlation between abrasion resistance and cold crushing strength. Therefore, as a rule of thumb, cold crushing strength can be used to predict abrasion resistance. And if an abrasion resistance must be met, a minimum cold crushing strength requirement could be derived from Fig. 14.

7 Recommendations for the standards

ISO 16282:2007-10 does not require glass plate calibration and states that precision data are not available. On the other hand, ASTM C704 states that the calibration is a supplementary requirement. However, this interlaboratory study would strongly recommend to include the precision data obtained in this study into ISO 16282:~~2008.~~ Furthermore, it strongly advises the use of a glass plate for calibration and to adjust the air pressure to fit the target 9,3 cm³ ± 0,3 cm³ abrasion of the glass piece. Moreover, when taking these steps into consideration, the use of tubes other than glass ones could be accepted if the target values of calibration are met.

If the abrasion resistance is higher than, for instance 15 cm³, it should be allowed to reduce the amount of abrasive medium (SiC grains) by 50 % and to just record double the result obtained as abrasion resistance, this would prevent the specimen from breaking during testing.

Since refractories are used at high temperatures (>1000 °C) further investigations should focus on hot abrasion resistance. Such temperatures will bear strong effects on refractories microstructure. At high temperature, softening, appearance of highly viscous phases, development of stresses due to thermal expansion mismatch of different aggregates, etc., will bear a clear influence on abrasion resistance.

References

[1] ISO 16282:2007-10 Methods of test for dense shaped refractory products — Determination of resistance to abrasion at ambient temperature

[2] ASTM C704/C704M-15 Standard Test Method for Abrasion Resistance of Refractory Materials at Room Temperature

[3] ISO 16349:2015-01 Refractory materials - Determination of abrasion resistance at elevated temperature

[4] Urbanek, G.; Klischat, H.-J.; Miranda-Martinez, M.: Evaluation of Test Methods for Refractory Bricks: A comparison and Validity Study of some Cold Crushing Strength Standards. Part 1. refractories WORLDFORUM 15 (2023) Nr. 2, p. 56-67

[5] Urbanek, G.; Klischat, H.-J.; Miranda-Martinez, M.: Evaluation of Test Methods for Refractory Bricks: A comparison and Validity Study of some Cold Crushing Strength Standards. Part 2. Refractories WORLDFORUM 15 (2023) Nr. 3, p. 55–63

[6] Urbanek, G.; Klischat, H.-J.; Miranda-Martinez, M.: Evaluation of Test Methods for Refractory Bricks: A comparison and Validity Study of some Cold Crushing Strength Standards. Part 3. Refractories WORLDFORUM 15 (2023) Nr. 4, p.

[7] Krause, O.; Urbanek, G.; Körber, H.: Determining Resistance to Abrasion at Ambient Temperature – Improving Comparability Between Laboratories. RHI Bulletin 2013, Nr. 2, pp. 44-49

[8] Krause, O.; Urbanek, G.; Körber, H.: Determining Resistance to Abrasion at Ambient Temperature –in Order to Establish Better Comparability Between Laboratories. Refractories WORLDFORUM 5 (2013) No. 2, pp. 59–64

[9] ISO 5725-2: 2019-2:2019-12 Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

[10] ASTM E691:2022 Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

[11] Routschka, G.; Wuthnow, H. (eds.), Handbook of Refractory Materials, 4th ed., Vulkan-Verlag, Essen 2012, p. 221–222

[12] ISO 10059-1:1992-02 Dense, shaped refractory products; determination of cold compressive strength; part 1: referee test without packing

[13] ASTM C133-97 (reapproved 2021) Cold Crushing Strength and Modulus of Rupture of Refractories

[14] Clavaud, B.; Martin-Calle, S.; Mathia, T.: Abrasion – Experimental approach: An Attempt to take into Account Field Conditions. Proceedings UNITECR 93 (in conjunction with XXII ALAFAR Congress), Sao Paulo, pp. 1550–1561