Determination of Resistance to Abrasion at Ambient Temperature – in Order to Establish Better Comparability Between Laboratories, Existing Standards Have to Be Revised

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In order to gain a deeper understanding how highly accelerated particles affect the surface of a refractory material during service, two test methods, ASTM C 704 and EN ISO 16282 were established. In the past imprecise data obtained by these methods lead to unjustified complaints of the refractory user industry when a third party laboratory cross checked the specified values for the abrasion resistance. The economic impact for the refractory producers as well as for the consumers is tremendous and painful. In order to enhance the reproducibility of this test, the latest revisions of ASTM C 704 in 2009 and 2012 result in a more rigid definition of the testing device. However the authors propose a different route to perform blast abrasion tests which generate highly reproducible abrasion values even if the tests are performed in different laboratories. The key to obtain rigid test values is that prior to the test the pressure supply for blast gun has to be adjusted till the abrasion volume for a standard float glass sample yields the exact value of 9,3 cm^3 ± 0,3 %. The properties of the standard float glass plates are defined in ASTM C 704-12. As a consequence it is proposed that the standard air pressure should no longer be defined at 4,5 bar but shall be adjustable.
The effectiveness of this measure was constrained by a round robin test that was performed in accordance to ISO 5725 in 2012. In this test 10 laboratories were involved and 6 different refractory materials were tested. The tests were performed according to ASTM C 704-09 with a standard air pressure of 4,5 bar. A second test series was conducted in which the test conditions were modified that way that the air pressure was adjusted till the abrasion result for a standard float glass sample yields the exact value of 9,3 cm^3 ± 0,3 %. The results of both test runs were compared with each other and with the precision date published in ASTM C 704-09 and ASTM C 704-12 (latest revision).
The major outcome of the round robin test is that the precision of results can be significantly enhanced if the air pressure is calibrated prior to the test by using a standard float glass sample. Due to the wear inside the venturi system it is necessary to readjust the air pressure with float glass samples frequently. It is recommended to do this every 20 tests. If the air pressure is adjusted properly the detailed layout of the apparatus dimension has no significant influence on the precision of the results.
The gained results are robust enough to recommend a revision of the actual EN ISO 16282.

1 Introduction

The resistance of refractory materials against particle abrasion is a key issue for many industrial furnaces where particle-loaded gas jets are expected in the process. They occur e.g. in furnaces of the petrochemical industry, waste incineration, coal-fired power plants and pusher furnaces for the second heat of steel.
In the past two test methods for the evaluation of the abrasion resistance have been established, namely ASTM C 704-12 firstly published in 1972 and EN ISO 16282 published in 2008. More precisely EN ISO 16282 is derived from the older ASTM C 704-01 with minor editorial and technical changes. Both methods describe almost the same test procedure that leads to reliable and comparable results within a single laboratory. However, if values are compared between different laboratories it turns obvious that the obtained values

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show significant and systematic deviations. This context is envisaged in Tab. 2 (ASTM C704 standard method), where the relative reproducibility % R is reported between 45 and 85 % depending to the refractory material. To counter the said imprecision, the ASTM committee C08 revised ASTM C 704-01 and published ASTM C 704-09. In this issue the dimension of the blast gun and the venturi system was defined in more detail and a new clause was added which describes known factors that affect the results. The findings in this clause base on a ruggedness test using float glass samples. Statistically significant effects are reported if the nozzle tube inside diameter and the air pressure are not precisely adjusted as defined in the standard. The latest revision of ASTM 704 dates in the year 2012 and was published in 2013. In this version again a precision of apparatus setup was defined. Supplementary requirements for highly abrasion resistant materials were added. The major task of this clause is to define more precise testing conditions that are ascertained e.g. by mounting additional pressure gauges. It is obvious that the ASTM committee C08 is heading toward a more rigid definition of the apparatus dimensions especially the particle acceleration system.

The material loss, typically calculated as volume loss, basically is dependent on the impact energy that is discharged when highly accelerated SiC particles hit the surface of the sample. It is proportional to the particle velocity and the weight (grain size) of the particle. Therefore only narrow SiC grain distributions are suitable for the test. It is even necessary to examine the grain size distribution within one batch if disintegration effects are obvious. In further the particle surface plays a role. The particles should have angular, jagged edged surfaces and therefore should be used only once. The bulk weight of the grains provides a possibility to check the grain shape in general.

Many factors affect the particle velocity during the acceleration. Of major importance is the standard air pressure provided for the venturi system. Both standards fix the value at 4,5 bar. However, the acceleration of the particles inside the blast gun is not only dependent on the pressure but also on the dimension of the chamber inside the blast gun and the distinct dimension of the venturi nozzle. The latter is described in detail in ASTM C 704 since the standard was revised in 2009. However, as long as ASTM C 704 is established, a simple but essential problem is discussed among non-US applicants of the standard. The blast gun as it is specified is not available in Europe. Moreover since 2009 ASTM stated the described blast gun as the only one which is permitted.

After the particles are accelerated in the venturi chamber, they have to pass a glass tube with strictly defined dimensions (length and diameter). Due to different surface roughness other materials to manufacture the tube may cause different friction and may decelerate the SiC grains differently during their passage through the tube. Some factors known to affect the results are already listed in ASTM C 704 since 2008 but without any quantification.

In summary the state of art is that existing standards tend to define the test conditions and the apparatus set-up more and more strictly in order to get a better inter-laboratory reproducibility. On the other hand it turns more difficult to keep the measurement device in the predefined condition and the method turns more error-prone. Therefore this paper envisages an alternative solution that is much easier to handle and leads to highly reliable results. It is proposed to deregulate the defined apparatus set-up and define a preliminary step to adjust the abrasion volume by measurement of a standard sample. Float glass specimens, described as calibration standard in ASTM C 704, qualifies as valuable standard as this material is already broadly in use for calibration purposes. This material is particularly suitable, because it is highly homogenous. To obtain a value of 9,3 cm^3 for this material, the standard air pressure is altered in disagreement to the standards. In principle it turned out that after the said adjustment the detailed apparatus set-up is of secondary importance. The results of the round robin test as described below clearly show that the said measures are suitable to provide reliable data for the blast wear of refractory materials.

2 Preliminary examinations

Prior to the round robin test investigations were undertaken to understand how variable air pressures and different amounts of abrasive material affect the results. In further an alternative material (B_4C) was used as nozzle tube. The preliminary tests were performed in two different laboratories.

2.1 Implication of the air pressure

In this preliminary test, borosilicate glass samples of a European producer with a density of 2,22 g/cm^3 were abraded at predefined air pressures between 3 and 6 bar. All other parameters were kept constant and in accordance to the definitions as described in ASTM C 704.

Fig. 1 shows that the volume loss is indicated by a linear ascending correlation over the measured range with increasing air pressure. However the measurements from both laboratories show a different slope. This indicates a slightly different set-up of the testing devices. If the nominal value of 7,4 cm^3 should be obtained the air pressure has to be adjusted individually to approx 5,50 for Laboratory A and 4,56 bar for Laboratory B.

2.2 Implication of the amount of abrasive material and the nozzle tube material

In this preliminary test float glass samples of a European producer and standard float glass samples as recommended by ASTM C 704 were abraded with different amounts of SiC (800 to 1200 g). Two sets of test were performed, one with the conventional glass nozzle tube and one with a B_4C nozzle tube. Each test was repeated two times. All other parameters were kept constant and in accordance to the definitions as described in ASTM C 704. The predefined air pressure therefore was 4,5 bar. All tests were performed on a single abrasion tester. First of all it can be stated that the abrasion loss correlates strictly linear over the measured range with the amount of SiC used in the test run (Fig. 2). Therefore the amount of SiC can also be used for the adjustment of the abrasion tester. In this case the mass of SiC has to be corrected till the measured value meets the standard value of 9,3 cm^3 as certified in ASTM C 704 for the reference float glass plates.

The linear correlation of the amount of SiC in use and the abrasion values encourages proposing that materials with a low abrasion resistance (>20 cm^3) may be tested with an alternative amount of SiC (e.g.

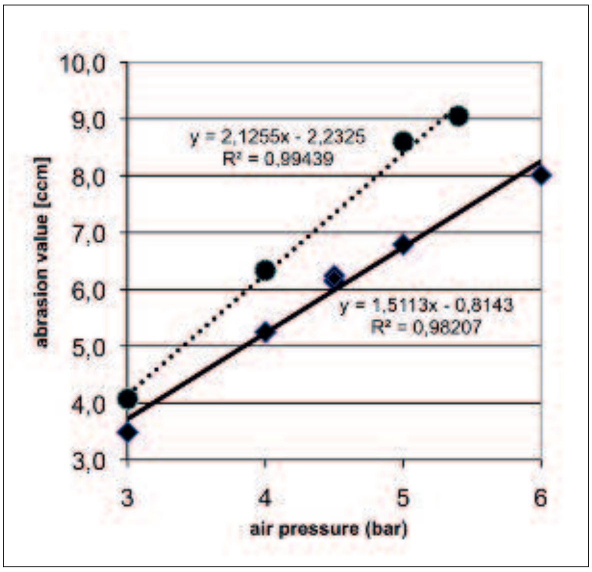

Fig. 1 Dependence of volume loss [cm³] and air pressure [bar] on borosilicate glass plates. The results are derived in two independent laboratories; the different signatures measurements in two different laboratories

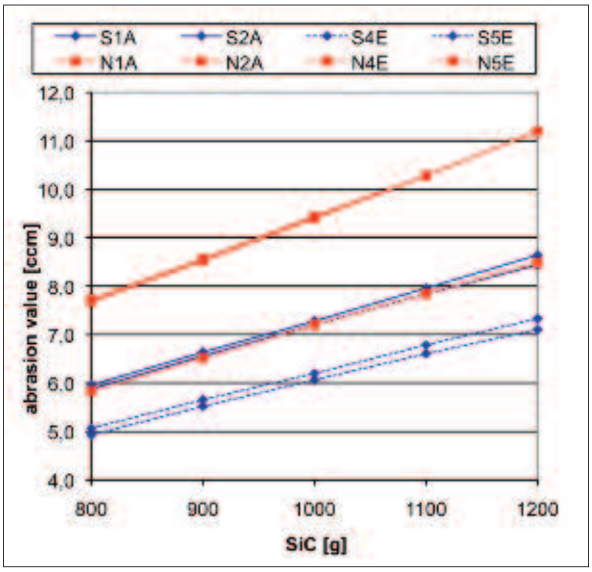

Fig. 2 Dependence of volume loss [cm³] and the amount of abrasive [g]. Two glass materials: the standard float glass sample (A) and a borosilicate glass (E) were tested. The blue lines represent measurements with the standard glass nozzle (diamonds) and the red lines were derived with B_4C nozzles (boxes). All conditions were measured twice; the replication measurements derived with B_4C nozzles plot very close together thus only a single regression line is visible

500 g instead of 1000 g). This will in any case reduce the danger to blast through the sample. The abrasion values obtained by using a different amount of SiC could be easily recalculated to values that stand for the abrasion when 1000 g SiC are used.

In further Fig. 2 clearly shows that the measured values change if alternative nozzle tube material is used. B_4C nozzles typically lead to higher abrasion loss than the conventional glass nozzles. However it is also evident that B_4C nozzles lead to a closer correlation of the single measurements. In Fig. 2 they plot along the same linear regression. The results obtained by using the glass tubes show variations up to 2 %. The higher abrasion resistance of the B_4C nozzle tubes obviously lead to a higher correlation of results. In contrast conventional glass tubes already show remarkable erosion after a single test and therefore change their performance already during the test and have to be changed every test. B_4C nozzles have the advantage that they can be used about 100 times because the erosion rate is very low. The air pressure of coarse has to be adjusted to a different value as for the glass tubes.

If the erosion pits are observed after the test, it became visible that the blast jet is slightly different. B_4C nozzles typically deliver deeper pits with a smaller eroded area at the surface. The blast jet appears to be better focussed. However the slopes of all tests are very similar. Therefore it can be stated that the nature of the blast jet is of minor importance for the reproducibility of the method if it is permitted to adjust the air pressure.

3 Interlaboratory study (round robin test)

As previously described, the performance and the reproducibility of the standard test method according to ASTM C 704 or ISO 16282 is virtually dependent on the particle size and velocity before they hit the sample surface. It is very difficult to provide precise test conditions as they are defined in ASTM C 704. The preliminary tests clearly show that the abrasion loss is significantly and systematically controlled by the air pressure, which is provided for the SiC particle acceleration in the Venturi system. The aim of the round robin test was to show the reproducibility of the test when performed according to ASTM C 704-09 and how the reproducibility is affected if air pressure is adjusted to an abrasion value of 9,3 g/cm³ which is certified for the standard ASTM float glass samples.

3.1 Set-up of the round robin test

For this round robin test 6 differently shaped and monolithic refractories were examined in 10 laboratories. The materials were selected with the aim to cover a wide

Tab. 1 Overview of the refractory materials considered for the interlaboratory test

Sample	Density [g/cm³]	Type of Refractory Product	Abrasion [cm³]
A	2,15	fireclay brick FC40	30
B	3,00	Alumina-chromia product type ACr80/5	15
C–D	3,15	Magnesia-chromite product type MCr50	7
E	3,00	Fired brick, based on sintermagnesia	15
F	2,20	Refractory concrete, conventional	8
G	2,80	Ultra low cement castable (ULCC)	5

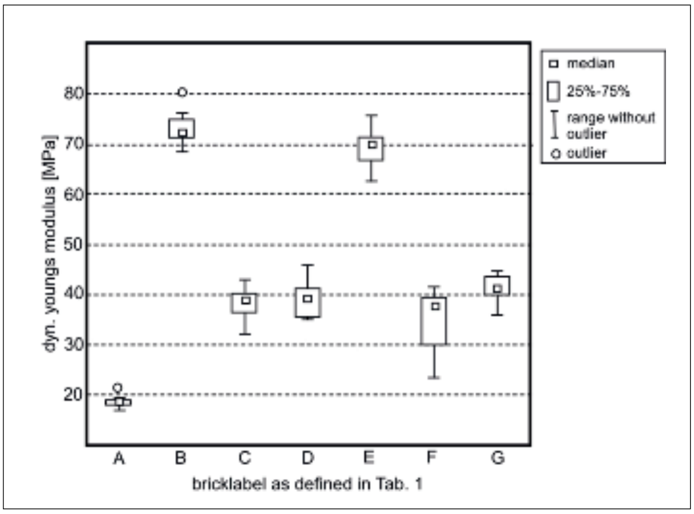

Fig. 3 **Box plot for the dynamic Youngs modulus derived from impulse excitation vibration technique. The results are shown for the six different refractory materials**

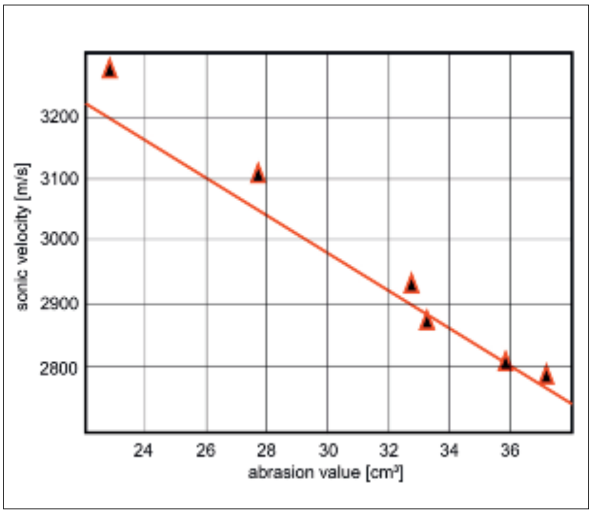

Fig. 4 **Linear dependence of the abrasion loss to the sonic velocity for sample A**

range of abrasion values (30 to 5 cm^3). Tab. 1 envisages the set of materials which was tested.

Prior to the round robin test all materials were prepared by moulding and cutting in order to meet the regulations defined in ASTM C 704 and ISO 16282. In order to screen the homogeneity of the selected sample specimens, they were examined by IEVT (impulse excitation vibration technique) and ultra sonic measurements in all three dimensions of the sample specimens. Fig. 3 documents the results of this preliminary test calculated as dynamic Youngs modulus. The results gained for the abrasion resistance are virtually dependent on the homogeneity of the provided sample specimens. As exemplified by sample series A in Fig. 4 the abrasion resistance is strongly dependent on the sonic velocity. A scatter of ≥10 % in sonic velocity was regarded as admissible for the round robin test.

As a preliminary result it may already be stated at this point of investigation that supplementary determination of the sonic velocity may be a powerful backing to evaluate the results of the abrasion resistance within a single refractory material, e.g. material A (Tab. 1).

Every laboratory was instructed to follow the same procedure. A first test run was performed with the standard ASTM float glass plates by applying the test condition as defined in ASTM C 704-09. The measurements were repeated twice with two glass specimens. The obtained results are important to evaluate the interlaboratory reproducibility at the state of art. In a second step, the laboratories were advised to run a first set of refractory material as they are reported in Tab. 1. The tests were performed under standard test conditions at fixed air pressure of 4,5 bar. Two measurements should be performed under the said conditions. The results are considered in the round robin test as values derived by the standard test conditions.

A second set of samples as shown in Tab. 1 were distributed among the laboratories to perform two further tests for all six different refractory materials. But prior to the test the adjustment of the pressure air was scheduled whereas the ASTM standard glass samples were used to calibrate the air pressure to attain an abrasion value of 9,3 cm^3 ± 0,3 %.

Finally 10 laboratories submitted 14 abrasion tests under standard conditions and 14 tests with adjusted air pressure. In further the standard glass plates were tested at least 4 times. Typically three glass plates were necessary to adjust the air pressure within the range of tolerance.

For the evaluation of the results the Software prolab plus *(Quodata Dresden/*DE*)* was used.

4 Results of the interlaboratory study (round robin test)

The results for the standard glass samples are reported first. The purpose for this study is to evaluate the impact on results if the air pressure is adjusted. After that the results for six refractory materials are presented. Two sets of results were determined where one set was measured with predefined and a second set with adjusted air pressure.

4.1 Results for standard glass samples after standard test procedure and adjusted

Fig. 5 shows the results for the standard ASTM float glass material. The mean value was fixed to 9,294 empirically and the tolerance limit was fixed to 8,620 and 9,992 cm^3 which corresponds to a Zu-value of <2,00. With a single exception, only values after adjustment fall within the tolerance limit. Within a single laboratory, the scatter of results are indicated by boxes. Laboratory 5 shows the broadest scatter. After cause analysis it turned out that this laboratory has distinct problems with a homogenous pressure air supply and could be solved by the installation of a pressure vessel.

In summary Fig. 6 clearly shows that that the adjustment of the air pressure significantly reduces the scatter of results.

4.2 Results for the refractory materials after standard test procedure and adjusted

Tab. 2 summarises the results of the round robin test. It can be clearly shown that the adjustment procedure described in this paper only slightly enhances the repeatability (Sr) within laboratories, but significantly enhances the standard deviation between

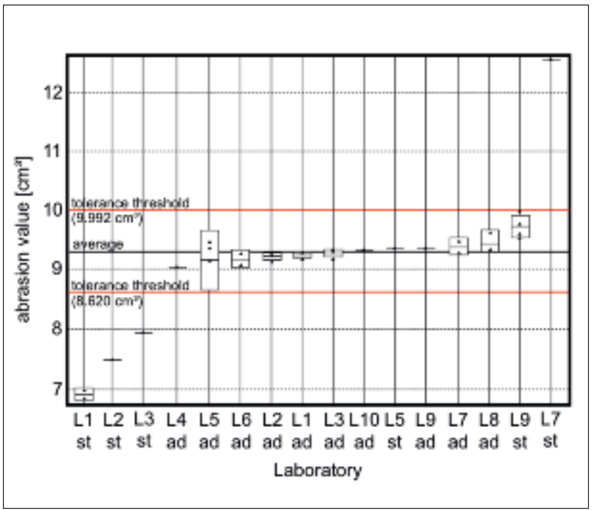

Fig. 5 Evaluation of the precision for the standard glass samples. The figure shows results for a fixed air pressure (st) and adjusted air pressure (ad). All values derived after adjustment fall within the tolerance threshold

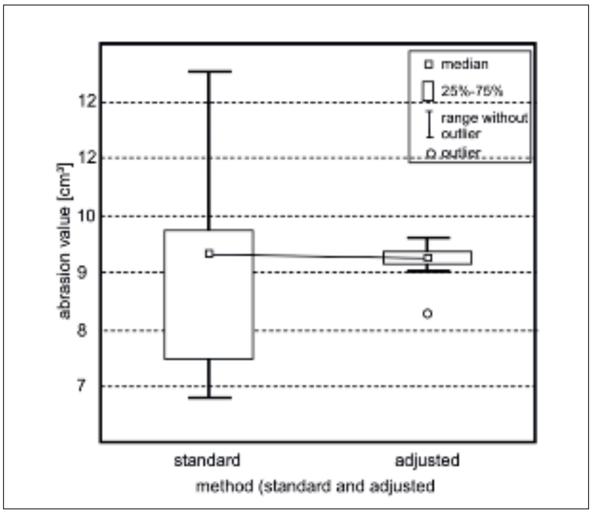

Fig. 6 Box plot for all standard glass sample measurements. The standard deviation can be significantly reduced if the air pressure is adjusted

laboratories (SR). Over all tested samples the average relative standard deviation between laboratories could be more than halved from 31,48 to 14,81 %. Sr is almost constant (8,19 to 7,54 %). It clearly shows that the abrasion testers are capable to produce reliable and consistent results. However, marginal differences in the dimensional setup, especially of the venturi design, lead to a poor accuracy of the results.

If only the results of the standard glass plates are considered the enhancement of the method by adjusting the air pressure even gets clearer. While the relative standard deviation between laboratories is improved by a factor 2, the glass plates really show room for improvement, because the glass samples are highly homogenous. Thus SR is decreased from 28,25 to 1,72 %, which denotes an improvement by a factor of almost 20. This clearly indicates that the adjustment method has a much higher precision than necessary if the heterogeneity of typical refractory material is considered. However a much higher repeatability is attainable if more test runs for a single material are performed. Sr is improved by $n^{-1/2}$, where n is the number of measurements.

In comparison to the round robin test that is published in the ASTM C 704, the adjustment method, as reported here, is in advantage if the average relative standard deviation between laboratories (SR_{ASTM} = 22,51 %) is considered. With 16,27 % as it is reported in ASTM C 704 SR is still 10 times higher than for the improved method reported here.

5 Conclusions

The presented round robin test results cover a broad range of shaped and unshaped materials with diverse blast abrasion resistances (from 5 to 30 cm³). The major outcome of this work is that it is better to adjust the air pressure of the Venturi system instead of defining more rigid regulations for the apparatus setup as recommended by ASTM C 704-12. If the air pressure is frequently adjusted by standard float

Tab. 2 Results of the round robin test. The detailed sample identification is listed in Tab. 1

	Sample	Average [cm³]	SR [%]	Sr [%]	R [%]	r [%]
Abrasion-resistance adjusted (ad)	A	33,911	7,05	2,52	19,74	7,06
	B	12,872	23,31	23,31	65,27	65,27
	C	7,72	9,45	6,57	26,46	18,4
	D	6,93	19,86	1,82	55,6	5,08
	E	12,841	9,18	5,47	25,71	15,32
	F_MOLD	8,56	22,01	7,94	61,63	22,24
	G_MOLD	5,304	23,85	4,31	66,78	12,06
	GLASS	9,266	1,72	1,57	4,82	4,4
	av		14,81	7,54	41,46	21,1

	Sample	Average [cm³]	SR [%]	Sr [%]	R [%]	r [%]
Abrasion-resistance standard (st) according to ASTM C 704 (fixed air pressure)	A	30,857	14,11	11,9	39,51	33,31
	B	14,518	26,72	26,72	74,81	74,81
	C	7,902	62,65	7,96	175,42	22,29
	D	7,62	34,9	2,48	97,72	6,93
	E	13,859	31,5	5,73	88,2	16,04
	F_MOLD	6,284	28,25		79,1	
	G_MOLD	5,054	30,74		86,06	
	GLASS	8,998	28,25	1,87	79,1	5,24
	av		31,48	8,19	88,15	22,92

glass samples the standard deviation between laboratories is reduced by the factor 2 if real refractory material is tested (SR decreases from 31,48 % to 14,81 %) and by the factor 20 (SR decreases from 28,25 to 1,72) if only the results of the standard glass samples are considered. The poorer precision of the refractory material can be delimited by the heterogeneity as typical for refractory materials. Sonic velocity measurements prior to the abrasion can help to identify outlier samples and SR can be positively affected by multiple measurements due to an enhancement of the repeatability. Over the measured range, the abrasion loss of refractory materials correlates linear with the air pressure and the amount of abrasive that was used. Therefore these parameters can be adjusted in a broad range without a significant drop of the precision of the method.

However it is important to note that the air pressure supply should be checked carefully. Temporary pressure fluctuations caused by an insufficient air supply will cause a poor repeatability and deliver in most cases lower values. Therefore it is necessary to confirm the pressure supply to be constant during the test.

The linear correlation of the amount of SiC in use and the abrasion values encourages to propose that materials with a low abrasion resistance (>20 cm³) may be tested with an alternative amount of SiC (e.g. 500 g instead of 1000 g). This will in any case reduce the danger to blast through the sample and will avoid unintended swirling of the SiC-particles in deeper holes. The abrasion values obtained by using a different amount of SiC could easily be recalculated to values that stand for the abrasion with 1000 g SiC.

In further it turned out that abrasion resistant materials for the design of the Venturi nozzle is beneficial for constant working conditions during the test. If the air pressure is adjusted all materials that are highly abrasion resistant can be taken in consideration. Nozzles made of B_4C can be highly recommended.

The findings of this study will be progressed for discussion to the national and international standardization boards. Changes to the current version of ISO 16282 will be proposed and discussed in ISO/TC 33 and CEN/TC 187 during the outstanding meetings this year.