Investigation of the Testing Parameters Influencing the Cold Crushing Strength Testing Results of Refractory Materials

J. Czechowski, A. Gerle, J. Podwórny, E. Dahlem

Compressive strength is commonly used for characterising the properties of refractory materials. There are a number of standards, which specify the methodology applied for testing dense shaped materials, insulating materials and unshaped materials. They contain requirements regarding the shape and size, the dimensional tolerance of test pieces, the manner of their preparation, the load rate and preload applied, the hardness and roughness of loading plates as well as the use of packing. These testing parameters are not fully defined in all the standards. Moreover, there are testing parameters not defined in standards, which can influence the obtained results of cold crushing strength (CCS).

The article presents investigations conducted within the framework of ReStaR project, aimed at determining the influence of testing parameters related to the CCS determination procedure on the testing results. In order to identify testing parameters that have the greatest influence on CCS determination in the case of dense formed, insulating and unshaped materials, the factorial design and variance analysis methods were applied. For each of these materials, each of the influencing factors on two adopted levels was examined.

It was found that a considerable impact on the determination result was exerted by the quality of preparing the test pieces, their format, in particular the application of preload and load rate in the case of unshaped products. It should be noted that the influence of the last one is different for dense products and insulating ones. The application of packing exerts a strong effect on a decrease of CCS value. In case of dense and insulating materials for factors chosen after preliminary tests, it was found that none of the tested factors is important if the results obtained in four laboratories are treated as one population, whereas for each laboratory the important factors were different. It means that the amount of experiments determines the quality of the test results. In case of unshaped materials, preparation and format of test pieces plays a key role for CCS testing results.

1 Introduction

Cold crushing strength (CCS) is a commonly used test for characterising refractory materials' mechanical strength at room temperature, which is always given in product datasheets. It is determined by a uniaxial compression test and calculated from the maximum force that a test piece can withstand before failure divided by the area of the loaded surface. Refractory products, as brittle and inhomogeneous materials, vary from one specimen to another. Moreover, the preparation of test specimens and the testing conditions can influence the test results and their dispersion. Different standards describe procedures of CCS testing for dense shaped refractories (EN 993-5, ISO 10059-1, ISO 10059-2 and ASTM C133), heat insulating products (EN ISO 8895 and ASTM C133), and unshaped products (EN ISO 1927-6 and ASTM C133). According to the existing standards for each group of materials, different requirements apply to the shape and size, dimensional tolerance, method of sample preparation, loading rate, preload, hardness and roughness of loading plates and packing. The above requirements are not fully defined in all the standards. There are parameters, which have not been described in the testing standards e.g. the casting direction and grinding for unshaped products. As a result, the influencing testing parameters related to the CCS test may

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Tab. 1 Factor levels for dense refractory materials

No.	Factor	Label	High Level (+)	Low Level (–)
X_1	Shape	Sh	cube	Cylinder
X_2	Extraction	Ex	corner	Middle
X_3	Pressing direction	Pd	//	⊥
X_4	Load rate	Lor	1,0 MPa	0,2 MPa
X_5	Preload	Prel	2000 Pa	No
X_6	Grinding	Gr	Yes	No
X_7	Height	H	36 mm	50 mm
X_8	Dimensions	D	36 mm	ø 50 mm
X_9	Parallelism	Para	//	Δh = 0,5 mm
X_{10}	Perpendicularity	Per	⊥	Δl = 2 mm
X_{11}	Roughness of plates	Rou	Ra 3,0–3,2 µm	Polished
X_{12}	Hardness of plates	Har	60 HRC	50 HRC
X_{13}	Packing	Pack	No	7 mm
X_{14}	Materials	Mat	A	C

vary in different laboratories and affect the obtained results.
Therefore, the major task is to identify the most significant parameters connected with the testing procedure, which influence CCS measurement results. The design of experiments and variance analysis in the presented studies were applied to determine the most significant testing parameters (factors), which affect the accuracy and precision of CCS testing (phase 1 of the ReStaR project [1]) of dense shaped, heat insulating and unshaped refractory products. Plackett-Burman design [2] was used because of its ability to estimate all main effects with the same precision and possibility of minimising the number of experiments. The main effect of each response is evaluated as the difference between averages of measurements made both at the high level (+1) and the low level (–1) of each factor. In this stage of the investigation, a large number of influencing factors were tested in one laboratory. In the second stage of the investigation (ReStaR phase 2), a limited number of significant testing parameters (factors) were analysed in interlaboratory tests.

2 Dense shaped refractory products

2.1 Materials and methodology

Shaped materials grade HA75 (High Alumina >75 % Al_2O_3), characterized by high strength, and grade MC95/10 (Magnesia Carbon >95 % MgO, ~10 % Carbon), characterized by low strength, both produced by RHI for the ReStaR research project, were tested. The homogeneity of test bricks was checked using an ultrasonic tester. The ultrasound velocity $(4,6 \pm 0,2) \cdot 10^3$ m/s for product HA75 and $(4,0 \pm 0,2) \cdot 10^3$ m/s for product MC95/10 were measured. Small dispersion of the results indicates the good homogeneity of both materials.
In the first stage of investigations fourteen relevant factors on two levels high (+) and low (–) were chosen (Tab. 1) for a Plackett-Burman factorial design.
According to EN 993-5 standard for dense materials, the lower and upper carrying steel plates should have hardness between 58 and 62 HRC, and their roughness Ra

Tab. 2 The Factorial Design Matrix for dense shaped refractory products and average CCS results from three tests obtained in each experiment; (+) corresponds to the upper level of the factor and (–) corresponds to the lower level of the factor

Exp.	Sh	Ex	Pd	Lr	Pl	Gr	H	D	Para	Per	Rou	Har	Pac	Mat	Average CCS ± SD [MPa]
	x_1	x_2	x_3	x_4	x_5	x_6	x_7	x_8	x_9	x_{10}	x_{11}	x_{12}	x_{13}	x_{14}	
1	+	+	+	–	–	–	+	–	–	–	+	+	+	–	39,7 ± 7,1
2	+	+	+	+	–	–	–	+	–	–	–	+	+	+	84,1 ± 19,7
3	–	+	+	+	+	–	–	–	+	–	–	–	+	+	90,9 ± 15,3
4	+	–	+	+	+	+	–	–	–	+	–	–	–	+	53,5 ± 11,3
5	+	+	–	+	+	+	+	–	–	–	+	–	–	–	35,5 ± 8,3
6	+	+	+	–	+	+	+	+	–	–	–	+	–	–	21,5 ± 4,3
7	–	+	+	+	–	+	+	+	+	–	–	–	+	–	40,1 ± 6,1
8	–	–	+	+	+	–	+	+	+	+	–	–	–	+	52,1 ± 11,8
9	–	–	–	+	+	+	–	+	+	+	+	–	–	–	33,9 ± 2,2
10	+	–	–	–	+	+	+	–	+	+	+	+	–	–	41,8 ± 2,8
11	–	+	–	–	–	+	+	+	–	+	+	+	+	–	34,2 ± 6,9
12	–	–	+	–	–	–	+	+	+	–	+	+	+	+	94,4 ± 21,3
13	–	–	–	+	–	–	–	+	+	+	–	+	+	+	102,9 ± 25,6
14	+	–	–	–	+	–	–	–	+	+	+	–	+	+	85,0 ± 17,6
15	+	+	–	–	–	+	–	–	–	+	+	+	–	+	66,0 ± 17,6
16	–	–	–	–	–	–	–	–	–	–	–	–	–	–	30,9 ± 4,6

should reach 0,8–3,2 µm. Plates with characteristics given in Tab. 1 were specially prepared for the tests. In the test with packing, a 7 mm-thick cellulose fibre wall-board was used.

The experimental design with 14 factors on two levels consisted of 16 experiments (ReStar phase 1). Each experiment was repeated three times, which means that 48 test pieces were tested.

2.2 Results and discussion

The regression equation was

$$y = 56,9\,x_o - 3,1\,x_1 - 4,9\,x_2 + 3,3\,x_3 + 4,5\,x_4 - 4,8\,x_5 - 15,6\,x_6 - 11,6\,x_7 + 1,6\,x_8 + 10,8\,x_9 + 1,9\,x_{10} - 3,2\,x_{11} + 4,4\,x_{12} + 14,6\,x_{13} + 21,8\,x_{14} \quad (1)$$

The critical value of t-student (t_{crit}) was 2,03. The determined levels of significance of equation regression coefficients (t_{cal}) on significance level $\alpha = 0,05$ (the number of the degrees of freedom f = 33) are presented in Fig. 1.

The obtained results presented in Fig. 1 revealed the following:

- Factors which have a strong influence on CCS results include: type of material (x_{14}), packing (x_{13}), test piece height (x_7), the parallelism of the loaded surfaces (x_9) and grinding (x_6);
- Extraction (x_2), load rate (x_4), preload (x_5) and hardness of plates (x_{12}) exerted only a minor influence;
- Close to t_{crit} are the calculated values of t_{cal} for such factors as shape (x_1), direction of pressing (x_3) and roughness of plates (x_{11}) (in the R_a range of 0–3,2 µm), so their influence is very small;
- Dimensions (x_8) and perpendicularity (x_{10}) in the tested range (with a tolerance of 2 mm) have a negligible influence on the CCS results.

On the basis of the conducted investigations, it was found that due to a strong influence of 7 mm thick cardboard spacers on the result of CCS determination, their use should be eliminated. To avoid the impact of sample preparation for tests, in particular the influence of surface non-parallelism, it is necessary to pay special attention to this element in the process of sample preparation before tests. As regards steel plates used as spacers, the conditions related to their hardness and roughness of surface defined in the current testing standard can be considered sufficient.

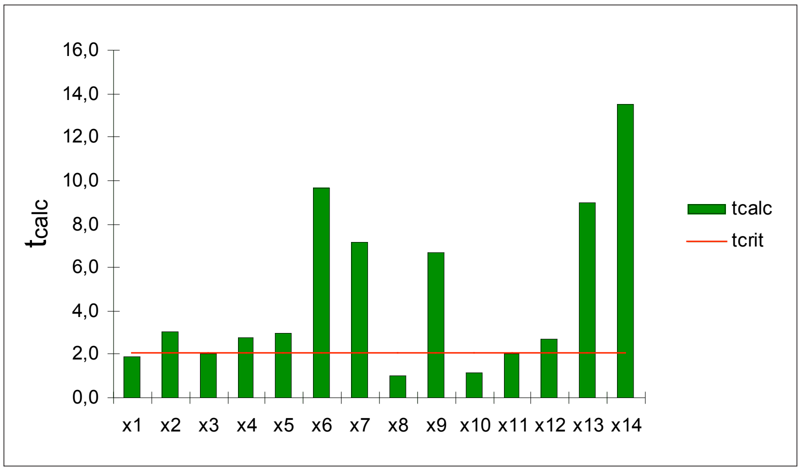

Fig. 1 **The CCS testing significance factors for dense shaped materials**

Taking into consideration the obtained results and conclusions formulated at the second stage, tests in four different laboratories were performed, while limiting the number of factors to four on two levels are presented in Tab. 3 (ReStaR phase 2).

Additionally, the „extraction" factor was taken into account by cutting the samples out of the centre and the outer part of the examined shapes.

Eight experiments, repeated three times in each of the four laboratories, were planned. In total 96 test pieces were examined. The summed up results from all the laboratories (Tab. 4) revealed differences in the results of CCS testing and in the standard deviations of the results (SD).

Maximum standard deviation reached 11,5 MPa. The highest values of standard deviations were observed in the case of test pieces in a form of cubes, irrespective of surface preparation and load rate.

The influence of material's non-homogeneity on the obtained results of compressive strength was analysed on the basis of results given in Tab. 4. The results obtained for test pieces cut out from the outer part (64 test pieces) and the ones cut out from the centre of the shapes (32 test pieces) were separated.

Higher values of CCS testing results, observed in the case of samples cut out from the shapes' centre (Tab. 5), indicate the examined material's heterogeneity. How-

Tab. 3 **Factors and levels chosen for CCS design of experiment in ReStaR phase 2**

Factor	High Level (+)	Low Level (–)
Shape	Cube	Cylinder
Geometry	36 mm × 36 mm	50 mm × 50 mm
Load rate	1,0 MPa/s	0,2 MPa/s
Grinding	Yes	No

Tab. 4 **A comparison of CCS testing results from four laboratories**

Exp.	Size [mm]	Grinding	Load Rate [MPa/s]	Format	CCS [MPa]	SD [MPa]
7	50 × 50	No	0,2	cylinder	58,9	4,6
1	50 × 50	No	1,0	cube	63,6	9,2
3	50 × 50	Yes	0,2	cube	65,4	10,1
8	50 × 50	Yes	1,0	cylinder	66,7	8,9
2	36 × 36	Yes	1,0	cube	62,1	11,5
4	36 × 36	Yes	0,2	cylinder	64,1	10,6
6	36 × 36	No	1,0	cylinder	61,1	8,4
5	36 × 36	No	0,2	cube	58,5	11,3

Tab. 5 Influence of sample extraction on CCS testing results

	CCS [MPa]	SD [MPa]
All samples	62,4	9,4
Samples from the outer part:		
Cylinders	62,1	8,3
Cubes	61,3	8,5
All samples from the outer part	61,9	8,4
Samples from the centre:		
Cylinders	62,8	8,7
Cubes	64,3	13,7
All samples from the centre	63,5	11,3

Tab. 6 Identified significant factors influencing the results obtained in particular laboratories

Laboratory	Signal Effect	Noise Effect
1	Grinding*Load rate	Grinding*Geometry
2	Grinding*Shape	Load rate
3	Shape*Load rate, Geometry*Shape	Geometry, Load rate
4	Grinding	Shape, Geometry

Tab. 7 Repeatability and reproducibility of CCS testing results obtained in different experiments

Run	7	1	3	8	2	4	6	5
Mean value, CCS [MPa]	58,9	63,6	65,4	66,7	62,1	64,1	61,1	58,5
Repeatability, s_r [MPa]	3,0	6,9	2,7	1,5	8,4	5,2	6,1	7,0
Repeatability, s_r [%]	5,1	10,8	4,1	2,2	13,6	8,0	9,9	12,0
Reproducibility, s_R [MPa]	4,9	9,7	11,1	9,5	12,0	11,4	8,8	12,0
Reproducibility, s_R [%]	8,3	15,2	16,9	14,3	19,4	17,9	14,5	20,6

ever, these differences are smaller for test pieces in a form of cylinders. In the case of cubes the CCS testing results are higher, particularly for test pieces extracted from the centre, where the dispersion of results is also considerably higher.

Tab. 6 shows factors influencing the results of CCS testing, determined on the basis of variance analysis in each of the laboratories.

The signal effect provides information about the major factors, whose level of statistical significance is $\alpha = 0,05$, whereas the noise effect represents the factors which have an impact on the standard deviation of the testing results.

The factor influencing the testing results – "Grinding" – was found only in laboratory 4. The remaining laboratories revealed significance of various mutual influences, which was most probably caused by an insufficient number of test pieces in one experiment in particular laboratories. When all results were treated on different way, it means as one population (instead of four presented in Tab. 6), it was found that none of the factors were significant. In this case also, no mutual influence of factors was observed to reach the level of significance $\alpha = 0,05$. Tab. 7 presents the computed values of repeatability (calculated as standard deviation of standard deviations obtained for each laboratory under repeatability conditions: the same laboratory, operator and equipment) and reproducibility (different laboratories, operators and equipment) for particular experiments.

In run 7, the values of repeatability and reproducibility of the testing results were found to be the lowest. However, the obtained CCS values were also very low. In run 3, the values obtained for CCS were the highest, repeatability was similar to that in run 7, but reproducibility was found to be twice higher. It is worth emphasizing that in run 7 the test pieces had a form of cylinders, whereas in run 3 they were cubes. Results of conducted F-test (probability 0,95) revealed that differences in values of repeatability and reproducibility between particular experiments are important. This entitles to determine the conditions of the run 7 as giving the best reproducibility which is especially important from the point of view of the objectives of the project.

3 Shaped insulating refractory products

3.1 Material and methodology

Insulating bricks containing ca. 30 % of Al_2O_3 and 60 % of SiO_2, having the dimensions of 230 mm × 114 mm × 64 mm and 230 mm × 114 mm × 76 mm, were subjected to CCS tests. Before the tests, the homogeneity of each test piece was checked by measuring the geometric density, which reached 0,536 ± 0,060 g/cm^3. The small dispersion of results suggested their homogeneity.

A factorial design matrix was developed for six selected factors on the levels quoted in Tab. 8. Each experiment was repeated three times, so the total number of samples prepared for tests was 24 (ReStaR phase 1).

Tab. 8 Factors and their levels in CCS tests of insulating materials

Exp.	Load Rate [MPa]	Preload [Pa]	Height [mm]	Dimensions [mm]	Packing [mm]	Operator	Average CCS ± SD [MPa]
	x_1	x_2	x_3	x_4	x_5	x_6	
1	0,2	No	64	114 × 114	No	A	2,1 ± 0,5
2	0,05	2000	64	230 × 114	No	A	1,8 ± 0,3
3	0,2	No	76	230 × 114	7	A	2,4 ± 0,3
4	0,2	2000	64	114 × 114	7	B	1,6 ± 0,2
5	0,2	2000	76	230 × 114	No	B	2,2 ± 0,6
6	0,05	2000	76	114 × 114	7	A	0,7 ± 0,1
7	0,05	No	76	114 × 114	No	B	2,0 ± 0,0
8	0,05	No	64	230 × 114	7	B	1,3 ± 0,1

3.2 Results

Based on the obtained results, the following regression equation was determined:

$y = 1{,}8\ x_0 + 0{,}3\ x_1 - 0{,}2\ x_2 + 0{,}06\ x_3 - 0{,}2\ x_4 + 0{,}3\ x_5 - 0{,}01\ x_6$

The critical t-student value (t_{crit}) was 2,11 on the significance level of $\alpha = 0{,}05$ (degrees of freedom f = 17).

Fig. 3 presents the calculated values of significance compared to the critical t-student value.

There is clearly no influence of the operator (x_6) and test piece height (x_3) on the obtained results: t_{cal} for x_6 and x_3 is much lower than the value of t_{crit}. A considerable influence has been noticed in the case of preload (x_2) and dimension (x_4), while load rate (x_1) and packing (x_5) have been found to have a very strong influence on the results obtained.

At the second stage (ReStaR phase 2), the investigations were limited to establishing the influence of three factors defined as significant, namely, size, height and load rate, on the previously adopted levels. The tests were conducted in 4 laboratories, 8 experiments repeated 3 times in each laboratory. In total 96 test pieces were examined.

The mean values of CCS testing results and their standard deviations in particular experiments, which have been listed in Tab. 9, showed diversity and lack of notable influence of any of the factors. The results of calculations regarding the impact of the analysed factors on CCS determination (Tab. 10) have revealed that in the case of two laboratories no factor reached the significance level of 0,05, and in the remaining two laboratories they were different: "Dimension" and "Height", respectively, and the same kind of mutual effects was observed: "Dimension" and "Load rate". In laboratories where the "signal effect" was not found, the "noise effect" was observed. When the results of tests from all the laboratories were pooled in one factorial experimental design, it was found that none of the factors were significant; also, no mutual influences on the significance level of $\alpha = 0{,}05$ were observed. Tab. 11 presents the calculated values of repeatability and reproducibility for particular experiments.

For Run 4, the values of repeatability and reproducibility of the testing results were the lowest. However, the obtained mean value of CCS was also one of the lowest. In the case of Run 2, the CCS value obtained was the highest, and repeatability and reproducibility reached a similar level. Results of conducted F-test (probability 0,95) revealed that differences in values of repeatability and reproducibility between particular experiments are not important from statistical point of view. However value of F-statistic for repeatability and reproducibility which differs between experiments about 0,2 MPa was very close (2,78) to critical F value (2,82). Regardless of this it can be concluded, that independent on the conditions of runs the obtained results characterized good repeatability and reproducibility.

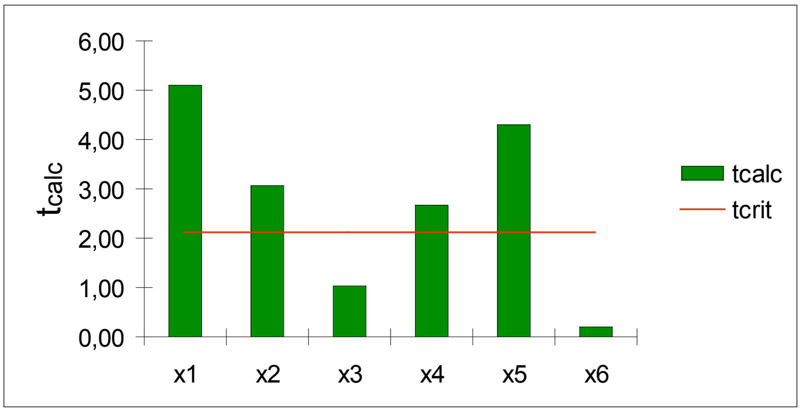

Fig. 2 The significance of factors influencing the CCS testing results of insulating materials

Tab. 9 A comparison of CCS testing design of experiment results involving four laboratories

Run	Dimension [mm]	Height	Load Rate [MPa/s]	CCS [MPa]	SD [MPa]
7	200 × 114	64	0,05	1,8	0,3
1	200 × 114	64	0,2	1,9	0,3
3	200 × 114	76	0,05	2,1	0,5
8	200 × 114	76	0,2	2,0	0,4
2	114 × 114	76	0,2	2,2	0,5
4	114 × 114	76	0,05	1,8	0,3
6	114 × 114	64	0,2	2,1	0,4
5	114 × 114	64	0,05	1,9	0,5

Tab. 10 The determined significant factors influencing the CCS testing result in particular laboratories

Laboratory	Signal Effect	Noise Effect
1	Dimension, Dimension*Load rate	No
2	No	Dimension*Shape
3	No	Height
4	Height, Dimension*Load rate	No

Tab. 11 Repeatability and reproducibility of CCS testing results obtained in different laboratories

Run	7	1	3	8	2	4	6	5
Mean value, CCS [MPa]	1,8	1,9	2,1	2,0	2,2	1,8	2,1	1,9
Repeatability, s_r [MPa]	0,3	0,3	0,5	0,3	0,5	0,3	0,4	0,3
Repeatability, s_r [%]	18,9	17,1	22,8	15,9	23,3	16,6	20,1	17,4
Reproducibility, s_R [MPa]	0,3	0,3	0,5	0,4	0,5	0,3	0,4	0,5
Reproducibility, s_R [%]	18,9	17,6	22,8	23,2	23,3	16,6	20,1	27,5

Tab. 12 Formats and preparation of samples from unshaped material for the 1st stage of CCS tests

Format	A, B, D	
Level	High (+)	Low (–)
Preparation	3 point bending	Cutting into 2 equal pieces by means of a saw

Tab. 13 Factors and levels for unshaped materials CCS testing (2nd stage)

No.	Factor	Label	High Level (+)	Low Level (–)
x_1	Preparation	Prep	3 point bending	Cutting into 2 equal pieces by means of a saw
x_2	Casting direction	Cdir	⊥	//
x_3	Load rate	Lor	1,0 MP/s	0,2 MPa/s
x_4	Preload	Prel	No	2000 Pa
x_5	Grinding	Gr	No	Yes
x_6	Packing	Pack	No	7 mm

Tab. 14 The results of CCS tests depending on the format and manner of sample preparation

Exp.	Format/Preparation Level	Average CCS ± SD [MPa]
1	A/+	93,7 ± 2,4
2	A/–	75,7 ± 2,6
3	B/+	92,1 ± 2,0
4	B/–	86,9 ± 7,9
5	D/+	90,6 ± 7,4
6	D/–	92,0 ± 4,9

4 Unshaped refractory products

4.1 Material and methodology

The tests were conducted for refractory medium-cement bauxite castable MCC75 (Medium Cement Castable, >75 % Al_2O_3) produced by Calderys for the needs of ReStaR project. According to the manufacturer's datasheet, the tested castable contained 78,0 mass-% of Al_2O_3, 15,0 mass-% of SiO_2, 3,1 mass-% of CaO and 1,0 mass-% of Fe_2O_3.

To the dry mix placed in a Hobart agitator, 8,8 % of municipal water having a temperature of ca. 20 °C was added, and, after mixing, the castable was cast into A, B and D moulds, the formats of which were 230 mm × 114 mm × 64 mm; 230 mm × 64 mm × 54 mm and 160 mm × 40 mm × 40 mm, respectively, depending on the designed experiments, and vibrated for 30 s. The test pieces were placed in a climate box (humidity of 90 %) for 48 h; next, after 24 h, the moulds were dismantled, the test pieces dried at 110 °C and compressive strength was examined after preparation according to the adopted design of experiments. Using an ultrasonic tester, the homogeneity of all the samples was examined. The average velocity of ultrasound was determined to $4,6 \cdot 10^3 \pm 0,3$ (10^3 m/s). The small dispersion of the results allowed treating them as homogenous.

In the first stage, the influence of the size and preparation of test pieces on the obtained results was examined. In order to do that, bars of castable in A, B and D formats were used, from which samples for CCS testing on two levels were prepared either by three-point bending or by cutting them into two pieces with a saw (Tab. 12). Compressive strength was tested according to EN ISO 1927-6, i.e. pressing perpendicular to the direction of casting, load rate of 1.0 MPa, without grinding, without preload and packing. Six determinations of CCS for each level were performed, i.e. a total of 36 test pieces were prepared.

In the second stage only one format C (230 mm × 64 mm × 64 mm) was applied and six factors on levels quoted in Tab. 13 were selected, for which a factorial design matrix was developed (ReStaR phase 1).

The tests were conducted in 6 experiments, with 3 repetitions. The total number of experiments was 48.

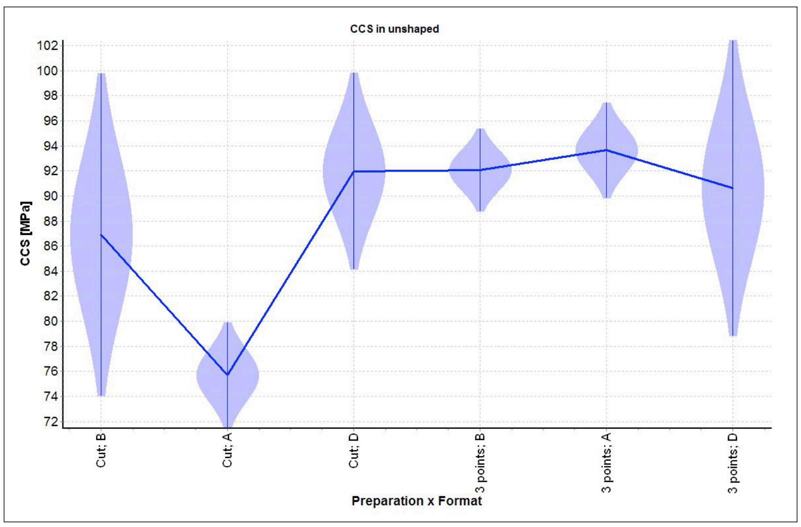

Fig. 3 Dependence of the predicted CCS value and the dispersion of results on the format (A, B or D) and manner of castable sample preparation (C – cut, 3pb – 3 point bending)

4.2 Results

The lowest values of CCS were obtained for the largest format A when the test pieces were prepared by cutting with a saw. For all test pieces that had been prepared by a previous 3-point bending test (i.e. subjected to MoR test) the average obtained values of CCS were similar, irrespective of the format. The plot presented in Fig. 5 shows that in the case of samples that had been cut from dried castables, one can expect a higher dispersion of results. The lowest dispersion of results was observed in the case of test pieces in A and B formats after having been subjected to a 3-point bending test, while the highest dispersion was observed in the case of "cut" samples in B format and "bent" samples in D format (Fig. 5).

For test pieces in D format, i.e. the smallest one, both cut and bent, a considerable dispersion of results was noticed.

Further investigations were conducted with test pieces in C format, taking into consideration the six factors quoted in Tab. 13, according to the adopted levels of variability. The factorial design matrix has been presented in Tab. 15.

On the basis of the obtained results, the following regression equation was determined:

$y = 68,5\ x_o + 3,8\ x_1 + 3,8\ x_2 + 3,1\ x_3 - 2,1\ x_4 - 0,04\ x_5 + 9,1\ x_6$

In Fig. 6 the significance of regression equation coefficients (t_{calc}) was compared to the determined value of $t_{crit} = 2,11$ (with the confidence level 0,95 and f = 17).

There is a notably strong influence of packing (x_6) on CCS results. A considerable impact is also exerted by the manner of sample preparation (x_1), load direction (x_2) in relation to the direction of casting as well as load rate (x_3). The value of tcal for preload (x_4) is similar to the value of tcrit, therefore, the influence of this factor can be treated as negligible, whereas grinding (x_5) has no effect at all.

An additional variance analysis revealed that interactions between x_4 and x_6 have a significant effect:

$y = 68,6 + 3,8\ x_1 + 3,8\ x_2 - 3,1\ x_3 - 2,1\ x_4 + 9,1\ x_6 + 3,8\ x_6 \cdot x_4$

The mutual interaction between packing and preload can be explained by pressing cellulose fibre paper under the applied preload.

For the third stage of tests (ReStaR phase 2), the castable samples were fired at 800 °C and three factors were selected: format, load rate and manner of sample preparation for tests. Each experiment was conducted in 5 laboratories and repeated 4 times, i.e. 20 test pieces were examined in each experiment. The tests were performed for a total of 160 samples.

The determined values of strength were higher compared to earlier tests because of the previous firing of the samples. The mean values and standard deviations in particular experiments are differentiated, but no evident factor was observed to influence these values. The results of calculations regarding the analysed factors' impact on CCS determinations (Tab. 17) indicated that particular laboratories have found a few main effects reaching the significance level of 0,05, among which "Format" and "Preparation" are the most common or occur as co-influences.

The differentiation and the character of „main effects" might be related to the material's non-homogeneity due to sample preparation and, in particular, the possibility of test pieces' rehydration in the process of wet cutting. For these reasons, it would be advisable to prepare samples by 3-point bending rather than cutting.

Tab. 15 **The factorial design matrix and average results corresponding to the experiments with given factors levels in CCS tests of an unshaped refractory castable**

Exp.	Preparation	Casting Direction	Load Rate [MPa/s]	Preload [Pa]	Grinding	Packing [mm]	Average CCS ± SD [MPa]
	x_1	x_2	x_3	x_4	x_5	x_6	
1	bending	//	0,2	No	No	No	82,4 ± 5,7
2	cutting	⊥	0,2	2000	No	No	79,1 ± 2,2
3	bending	//	1,0	2000	Yes	No	73,0 ± 7,9
4	bending	⊥	0,2	No	Yes	7	64,4 ± 2,7
5	bending	⊥	1,0	2000	No	7	70,0 ± 2,3
6	cutting	⊥	1,0	No	Yes	No	73,3 ± 1,9
7	cutting	//	1,0	No	No	7	42,9 ± 4,9
8	cutting	//	0,2	2000	Yes	7	61,0 ± 8,8

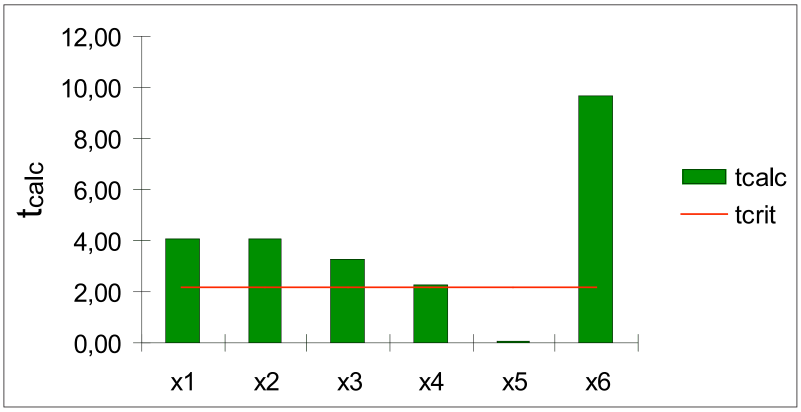

Fig. 4 **Significance of factors influencing the results of unshaped materials CCS testing**

Tab. 16 **A comparison of CCS testing results of an unshaped refractory product from five laboratories**

Exp.	Format	Load Rate [MPa/s]	Preparation	CCS [MPa]	SD [MPa]
7	D	1,0	cut	126,8	22,3
1	D	0,2	bending	108,4	16,4
3	B	1,0	bending	121,4	24,4
8	B	0,2	cut	112,6	23,6
2	B	0,2	bending	107,6	23,6
4	B	1,0	cut	129,0	19,9
6	D	0,2	cut	113,9	31,0
5	D	1,0	bending	110,0	22.3

Tab. 17 A list of computed significant factors for particular laboratories

Laboratory	Main Effect	Noise Effect
1	Load rate Format Preparation	No
2	Format Preparation Preparation *Format	Preparation
3	Preparation Format Preparation *Load rate	Format Preparation *Load rate
4	Load rate	Format
5	Format Format*Load rate Format* Preparation	No

Tab. 18 Significant factors influencing the CCS testing results for refractory castables as identified in the whole test with „Laboratory" considered as an additional factor

Main Effect	Noise Effect
Laboratory Preparation Format Laboratory*Format Format*Load rate Preparation*Format Laboratory*Load rate	Laboratory Laboratory*Format

Tab. 19 Reproducibility and repeatability of results in particular experiments

Experiment	7	1	3	8	2	4	6	5
Mean, CCS [MPa]	126,8	108,4	121,4	112,6	107,6	129,0	113,9	110,0
Repeatability, s_r [MPa]	8,2	2,6	5,6	12,7	7,8	8,9	13,1	3,7
Repeatability, s_r [%]	6,5	2,4	4,6	11,3	7,3	6,9	11,5	3,4
Reproducibility, s_R [MPa]	27,6	21,5	20,3	21,6	22,2	21,7	37,8	22,0
Reproducibility, s_R [%]	21,8	19,8	16,7	19,2	20,6	16,8	33,2	20,0

The inclusion of "Laboratory" in the group of factors influencing the result (factor "Laboratory") revealed that important factors were: "Laboratory", "Preparation" and "Format) (Tab. 18). These last two factors seem to confirm the significance of sample preparation for tests.

The analysis of repeatability and reproducibility of the CCS testing results revealed that the lowest value of repeatability and a low value of reproducibility was obtained in experiment 1 (Tab. 19), the value of CCS being one of the lowest in this test.

For reproducibility F-test revealed that only difference between the lowest value (20,3 MPa, experiment 3) and the highest value (37,8 MPa, experiment 6) are statistically significant. It was calculated that value of reproducibility greater about 13,8 from the smallest one (20,3, experiment 3) will be statistically important (34,1 MPa). In case of repeatability the value greater about 1,8 compare to the smallest one (2,6, experiment 1) will be statistically important (4,4 MPa).

5 Conclusions

- The factor which considerably influences the results of CCS testing is the quality of sample preparation for tests (parallelism and perpendicularity of the surface, its smoothness); it is, therefore, necessary to pay special attention to the manner the test pieces are prepared.
- Preload on the applied level causes a lower value of CCS testing results and a considerable dispersion of results.
- Load rate has an influence on the determined value of CCS testing results; it should be noted at this point that in the case of insulating materials the CCS value was found to be higher at a higher load rate (0,2 MPa/s), whereas in the case of dense shaped materials the CCS value was higher at a lower load rate (0,05 MPa/s).
- Application of packing strongly influences a decrease of the CCS testing results and the dispersion of results. The revealed negative effect of packing confirms earlier investigations described in literature [3].
- The statistical evaluations showed in case of dense and insulating products that none of the tested factors is important on investigated levels if the results obtained in 4 laboratories are treated as one population, whereas for each laboratory the important factors were different. It means that the amount of experiments determines the quality of the test results.
- For unshaped materials after drying, the obtained results strongly depend on the format and manner of test pieces preparation. The differentiation and character of "main effects" in case of fired material might be related to the material heterogeneity due to sample preparation and, in particular, the possibility of the test pieces rehydration in the process of wet cutting. For this reason it would be advisable using 3-point bending rather than cutting to prepare the test pieces.

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