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ANALYSIS AND QUALITY ASSESSMENT OF REFRACTORIES FORMED UNDER REDUCED PRESSURE

Refractories are the basic material for the construction of the lining of a melting furnace used, among other things, in the foundry industry. The article describes a comparative study of the influence of the type of moulding on the quality of the finished refractory product. A method for making products from refractory materials was proposed and a test methodology was developed. The results, based on a classic study of the quality of these materials, confirm a strong influence on the quality of the materials obtained in terms of reduced porosity and homogeneity of pore size.

Keywords: Foundry; melting furnace; refractory materials; vacuum moulding; porosity

1. Foreword

The requirements for the foundry industry are driving the development of new or upgraded manufacturing methods. Methods that are more efficient, precise and less time-consuming, with the main focus on high product quality, cost-effectiveness and environmental aspects. These new methods give the opportunity to meet the increasing requirements. Especially when are taking into consideration move complex casting products [9,12].

The casting production process is a multi-stage process with a high level of complexity. An additional challenge is the huge variety of parameters and their variability at each stage of casting production. These include:

- the multitude of casting materials and, consequently, their great variability in terms of parameters
- manufacturing methods and mould materials
- casting methods depending on the material and the size of the castings and their production per time unit

All of these elements results in searching of more efficient methods of obtaining the finished product at each stage of production [1,2,5,6,13,14,16,18].

One frequently used and universally applicable topic over time is the use of reduced pressure in various stages of casting production. This includes obtaining finished moulds after casting [19], the moulding sand compaction process – V process, Kuenkel-Wagner moulding machines, core making support, reduced pressure casting (1,2,6). Since the production of reduced pressure is neither a technologically difficult nor relatively expensive process and the effects achieved are at a satisfactory level [1,10], this article presents the results of research concerning the influence of reduced pressure on the quality parameters of refractory materials.

2. Introduction

Refractory materials are widely used in the foundry industry [3,4,7,17]. They are used both in the metal melting subsystem (as lining of furnaces, drain gutters, etc.) as well as in the forming subsystem (e.g. in the lost wax method) and in heat treatment furnaces. The materials have high requirements, concering high temperature resistance, no chemical reaction with the metal being melted and minimal thermal expansion. High mechanical properties (resistance of finished products to external and internal stresses), resistance to thermal shock (sudden temperature changes) and corrosion (resistance to slag and liquid metal) are also very important indicators [11,14,17].

Pores exert a dominating influence on the mechanical and thermomechanical properties of refractory material such as strength and thermal shock behavior. Thus, it is essential to characterize and quantify their number, size and size distribution. These parameters have a significant influence on the quality of the refractory materials obtained and thus on the quality of, for example, the lining in the melting furnaces, the purity of the

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metal, etc. The crucial indicator for most of these parameters is the final apparent density (total and open). It has a decisive and direct influence on the strength and slag resistance. If the density is higher, the above parameters have better (higher) values [12,17].

There are several methods of ensuring a sufficiently low level of porosity. The most common their parameter is the frequency of vibratory compaction and the duration of this process [3]. In this paper, the authors have proposed the use of reduced pressure moulding as a method to assist in obtaining a higher apparent density of the final product.

3. Materials and production

According to the Polish Standard PN-EN 993-12:2000 [20], aluminosilicate materials are refractory materials consisting, in different proportions, of two main components – SiO_2 and Al_2O_3. The main raw materials used are clay rocks in raw or roasted form, e.g. kaolinite group minerals ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$) with 39.5% Al_2O_3, 46.6% SiO_2 and 13.9% H_2O. In pure kaolinite, approximately 45% Al_2O_3 remains after roasting, creating a division of aluminosilicate materials into: chamotte – produced from typical clay raw materials, with an Asl_2O_3 content of up to 45%, and high-alumina, whose Al_2O_3 content exceeds 45%.

High alumino-silicate materials are characterised by a high alumina (III) content, above 45%. They can be manufactured from natural raw materials and semi-finished products. Natural raw materials include hydrated forms of aluminium (III) oxide such as hydragilite $Al_2O_3{\cdot}3H_2O$, bemite $Al_2O \cdot H_2O$ and diasporic $Al_2O_3{\cdot}H_2O$, and aluminium (III) silicates such as: andalusite, sillimanite, cyanite with anhydrous oxide formula $Al_2O_3 \cdot SiO_2$ and, less commonly, dumortierite $8Al_2O_3 \cdot 6SiO_2 \cdot B_2O_3 \cdot H_2O$ or topaz $Al_2(F, OH)2SiO_4$. Intermediates used in the production of high-alumina materials include technical alumina (III), fused and sintered alumina or mullite fuming [4].

The higher alumina (III) content makes it possible to obtain products with better strength and refractory properties, but natural raw materials are usually contaminated with harmful ferrous or calcareous admixtures, which results in use the enrichment and purification of the raw materials. If the impurities are not removed, the thermal properties can be drastically reduced.

From the raw materials supplied for testing, two groups were distinguished: first, raw materials and the intermediates, which had already been pre-cleaned and enriched.

The research methodology consists of:

1 – Pre-processing of raw materials
2 – Crushing and fractionation
3 – Mass preparation
4 – Drying
5 – Burning
6 – Cooling

Each of these stages has a separate specification to ensure that the quality of the intermediate product for the next process is maintained.

4. Technological properties of refractory materials and their significance

As a results of wide application of refractory materials and the high requirements given by the foundry industry, it is important to mention the basic parameters that characterise the materials [4,16].

a) The porosity of refractories is one of the most important parameters. It is directly related to the apparent density and determines, among other things, the corrosion phenomenon of the lining of melting furnaces, their slag resistance and their compressive or bending strength. A distinction is made between open and closed porosity. Open pores, especially through-pored pores, have a strong influence on slag penetration values, so it is important that this type of porosity is as small as possible, as a larger volume of open pores results in more rapid penetration of liquid slag into the material, e.g. the furnace lining, which will contribute to faster wear and tear and, as a result, increased plant operating costs and environmental pollution. Therefore, it is preferable to have a large number of small pores in the product, compared to the same volume of large pores [4,11,12].

b) the mechanical properties of the analysed materials determine the resistance of the finished products to external and internal stresses [3]. One such property is the compressive strength – CCS (from Cold Crushing Strength). Refractory materials used, for example, in blast furnace linings are primarily subject of compressive stresses [8]. Compressive strength is also related to the abrasion and impact strengths observed in industrial furnaces. In a sense, it is also a measure of durability in terms of the amount of load that the material can withstand in case of improper installation, e.g. if gaps between fittings are left too small, so that stresses are created between them due to thermal expansion of the material when the furnace is heated. The lower the compressive strength, the greater the chance that the lining will crack in case of an incorrectly built furnace [6].

c) The thermal properties of refractory materials are very important in terms of where they work, as these materials operate at high temperatures. One of the basic thermal properties is thermal expansion. Among other things, it affects the service life of the furnace lining. For higher expansion values, this must be taken into account by using larger expansion joints.

5. Laboratory studies

The aim of the study was to check the effect of moulding under reduced pressure on the properties of refractory materials. The material was compacted on a vibrating machine – in the traditional way and in a chamber with reduced pressure. The material used was high alumino-silicate [15]. Two blocks were moulded in one day – STD and VAC corresponding to moulding by vibration at atmospheric pressure and moulding by vibration using a vacuum pump, which resulted in a mould pressure of 4900 Pa.

The mould compaction methodology:

a) Preparation of two identical folding metal moulds. Lubrication with a release agent to facilitate removal after drying. The moulds were then assembled. One of them was inserted in a special sealed housing and a vacuum pump and vacuometer were connected to it,

b) preparation of the mass in a ZYKLOS mixer from a mixture of dry ingredients and water in proportions allowing to obtain 75 kg of ready mass with 6% humidity,

c) moulding on a vibrating table at a frequency of 60 Hz for 8 minutes (Fig. 1),

d) drying at 300°C, after drying the blocks were cooled in the air and removed from the moulds.

Fig. 1. Moulds on the vibrating table

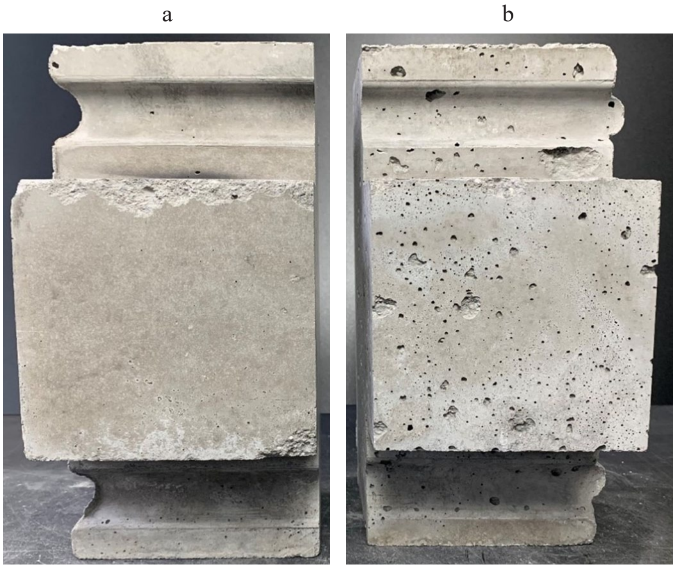
6. Analysis and evaluation

Tests were carried out on blocks obtained after forming the refractory material in moulds placed on a vibrating table. The blocks were cut in half to allow visual assessment and analysis of the number of pores inside the material under test.

6.1. Visual evaluation.

The visual assessment consisted of making an assessment of the visible pores on the side, top, bottom and cross-sectional surfaces through the centre of the block [15]. The next step was to measure the 15 largest pores on the sidewalls, front and rear walls and cross-sectional surfaces and compare their dimensions with each other. The measured surfaces are shown in Figs. 2 to 5 and the visualised calculations with diagrams in Figs. 6 to 9. No pores were measured for the top and bottom surfaces.

Optical analysis is one of the simplest and at the same time fastest methods to evaluate the effects of an experiment [3,5,14,15].



a b

Fig. 2. Comparison of the front surfaces of the blocks; a – VAC, b – STD

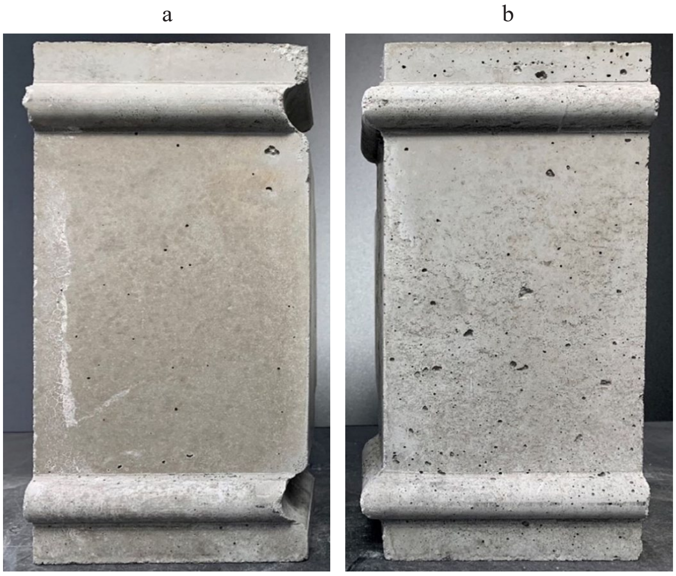


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Fig. 3. Comparison of the rear block surfaces; a – VAC, b – STD

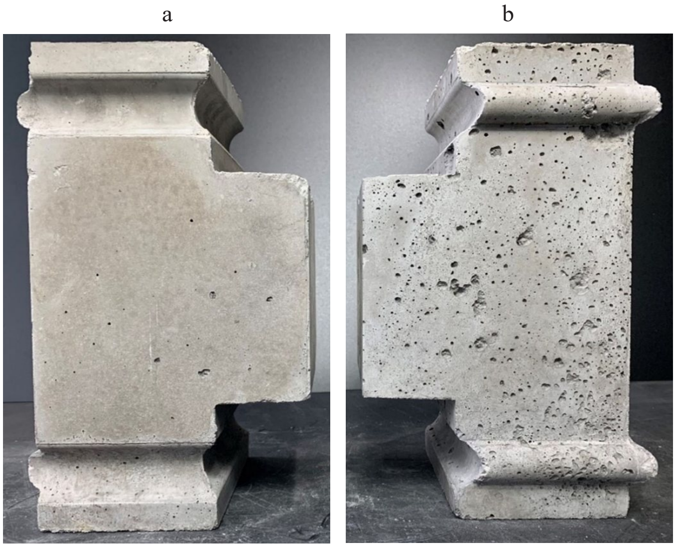

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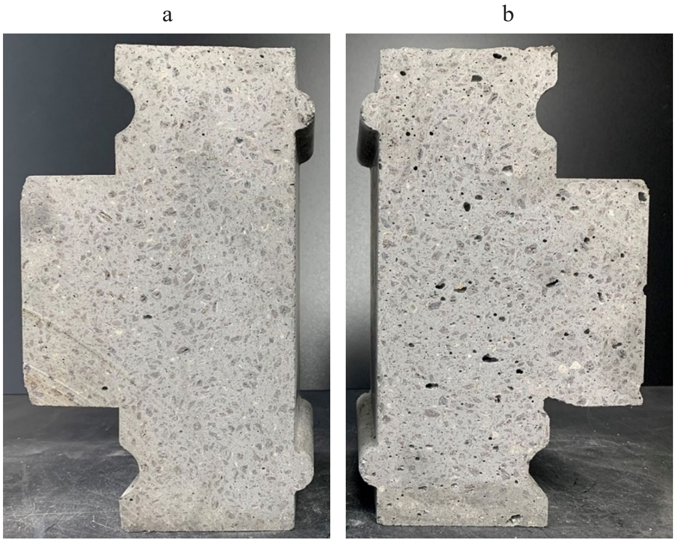
Fig. 4. Comparison of the lateral surfaces of the blocks; a – VAC, b – STD


a

b

Fig. 5. Comparison of cross-sectional areas of blocks; a – VAC, b – STD

Evaluating the images, it can be concluded that the results obtained from the compaction of the refractory material in the depressurised chamber are significantly better than in the material compacted by the traditional method. The porosity is significantly lower and the pores are smaller and less visible. The surfaces have same level and more homogeneous. Optical evaluation enabled a quick assessment of the effects of densification. For a more detailed analysis, the 15 largest pores were measured and their surface area calculated. The results obtained are shown in Figs. 6-9.

Summarising the results and the visual assessment analyses, the significant influence of the refractory forming method was clearly established, with a clear indication of the advantages of the reduced-pressure forming method. The visualisation in Figs. 6-9 and the calculation of the total pore area depending on the location and type of moulding in TABLE 1 clearly show the advantage of the vacuum method. The advantage ranges from a 4 to a 9 times reduction in pore area compared with the traditional method.

TABLE 1

Summary of pore areas for conventional and reduced-pressure moulding

Place of measure	**VAC**	**STD**	**Measure place**	**VAC**	**STD**
	Area [mm^2]			**Area [mm^2]**	
Front	252,9	1625,0	Sides	201,8	1986,3
Back	135,1	861,6	Cross-section	121,7	524,6

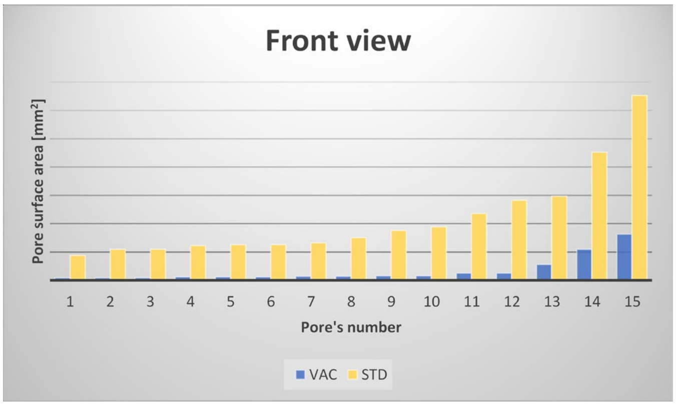


Fig. 6. Pore area – data from the front of the block

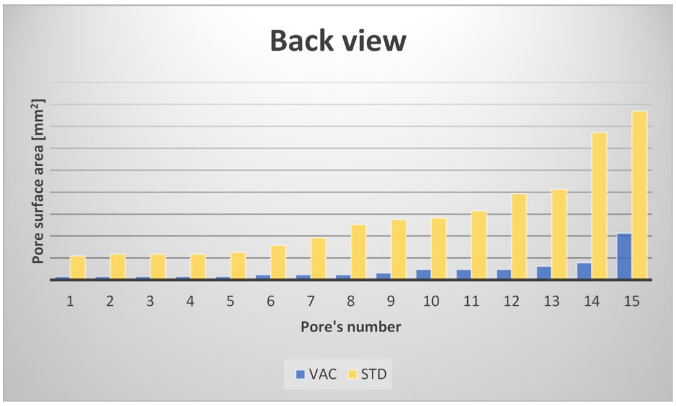


Fig. 7. Pore area – data from the back of the block

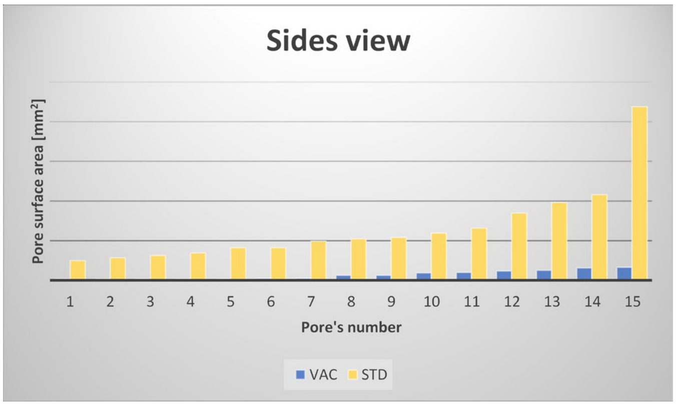


Fig. 8. Pore area – data from the side of the block

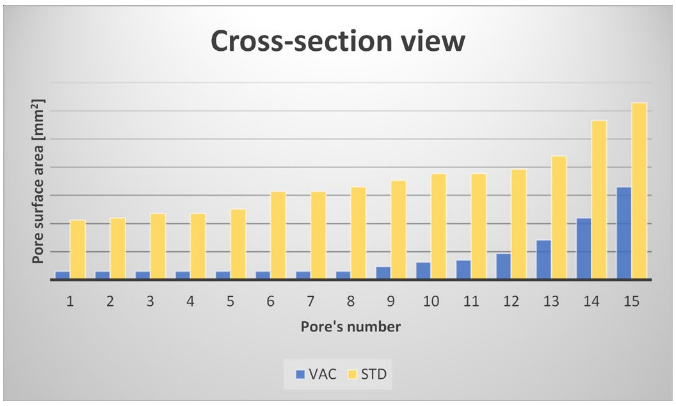


Fig. 9. Pore area – data from block cross-section

6.2. Microscopic analysis

Microscopic analysis confirmed the evaluation of the effect of moulding type on the quality (porosity) of the specimens [4]. The analysis was carried out using an optical microscope commonly used to assess this type of material and a scanning microscope [14].

Research methodology

Tests were prepared on pre-cut specimens using a precision saw, which were then dried for 1 hour at 110 degrees C. After this time, they were placed in a desiccator for cooling time. As a next step, Araldite DBF resin was mixed with Aradur Hardner HY 951 hardener at a ratio of 10:1 and inserted into a container connected to a vacuum pump to remove air bubbles from the resin. The specimens were inserted into the moulds with the test surfaces facing downwards and filled with the resin/hardener mixture. After filling the mould to approximately ¾ of its height, the sample was pressed against the bottom of the mould and set aside. This was repeated for four samples: STD1, STD2, VAC1, VAC2 [15]. The prepared samples were set aside in a vacuum vessel to remove air bubbles for 10 minutes. The samples were set aside for 24 hours for the resin to set.

The next step was to polish the samples using discs of 220 and 1200 gradations and a 3 μm polishing disc. For observation under the scanning electron microscope, the samples were additionally sputtered with tungsten and copper tape was applied to ensure that they were electrically conductive.

Optical microscope observations

The observation was carried out on all samples for magnifications of ×1, ×1.25, ×2. The resulting images are presented in Figs. 10-12 [15].

By analysing the structures in Figs. 9-11 obtained from optical microscopy observations, it can be seen that the VAC samples are less porous and that those pores that do occur are less circular in shape than those of the STD samples.

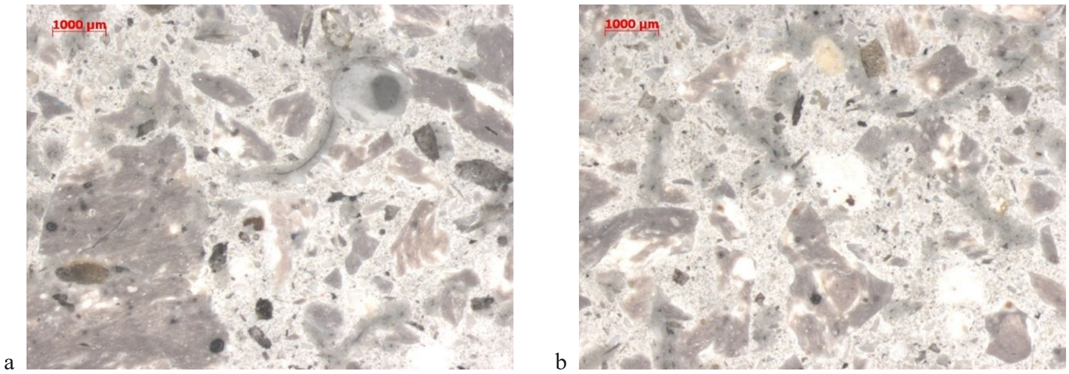


Fig. 10. Photo of the sample structure at ×1 magnification; a – STD1, b – VAC1

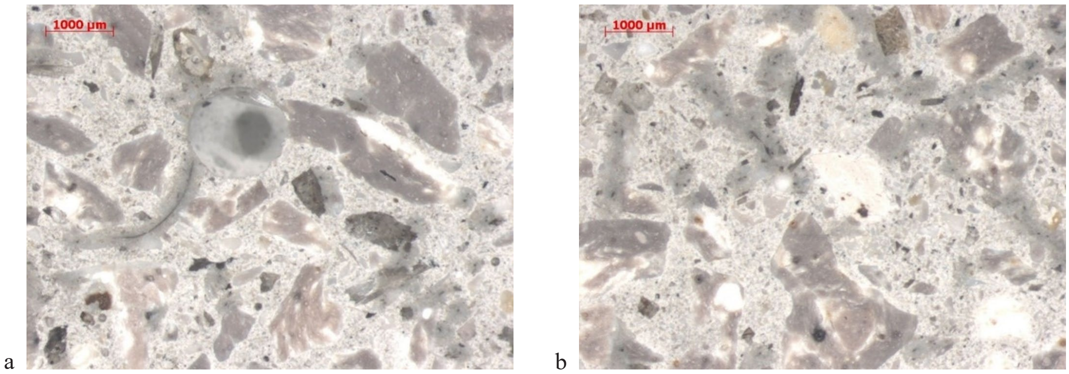


Fig. 11. Photograph of the sample structure at ×1.25 magnification; a – STD1, b – VAC1

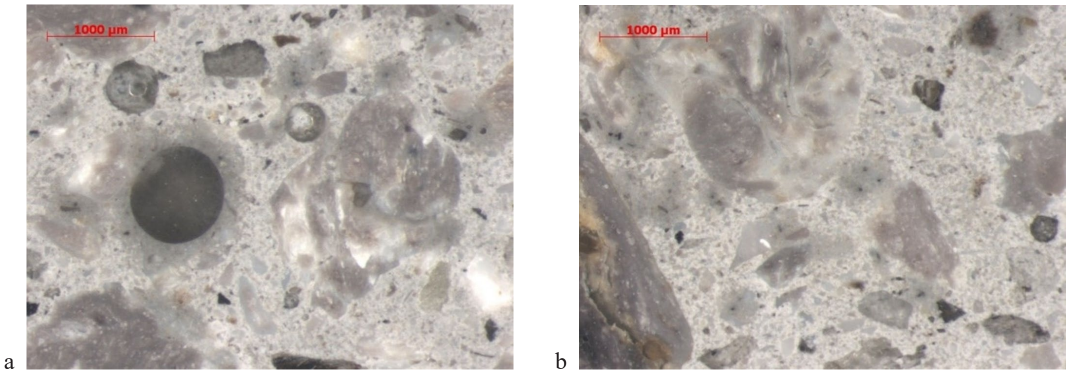


Fig. 12. Photo of the sample structure at ×2 magnification; a – STD1, b – VAC1

Electron microscopy (SEM) observations

Scanning electron microscope observations were carried out for two samples: STD1 and VAC1 at magnifications of 25×, 50× and 100×. The obtained images are visible in Figs. 13-15 [15].

The conclusions from the analysis of the SEM microscope images are the same with those of the optical microscope images. When observing the structures of the samples, clear differences in the number, size and shape of the pores were noted. The STD samples are characterised by a more porous structure, the spaces are larger and circular in shape. VAC samples have small pores with less regular shapes in much smaller numbers.

6.3. Strength test

The main task of refractory materials is to withstand the high temperature occurring f.e., foundry furnaces, ladles, etc., to

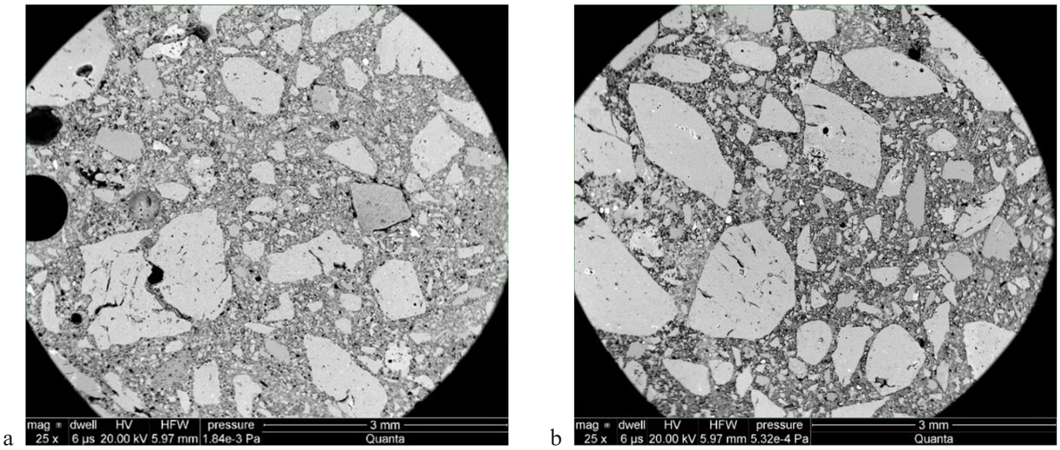


Fig. 13. Photograph of the sample structure at ×25 magnification; a – STD1, b – VAC1

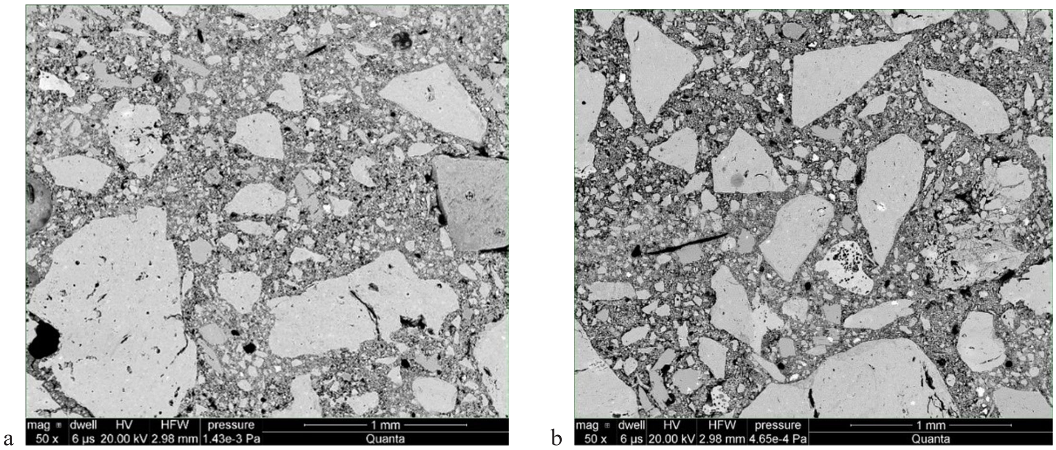


Fig. 14. Photograph of the sample structure at ×50 magnification; a – STD1, b – VAC1

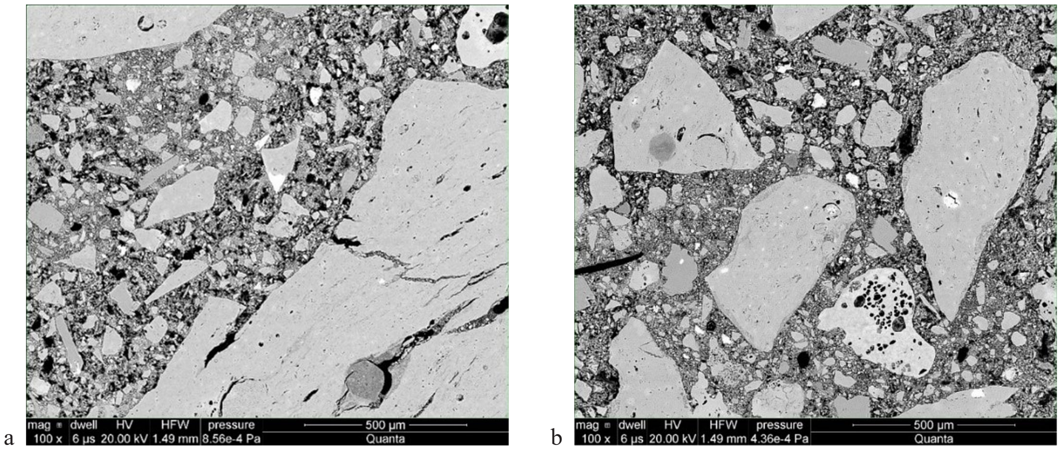


Fig. 15. Photograph of the sample structure at ×100 magnification; a – STD1, b – VAC1

prevent damage to other materials and to accumulate and retain heat where it is needed. One of the most important parameters determining the quality of the materials in question is the compressive strength [4]. The parameters of high alumina materials are defined, among others, by the Polish standard PN-EN 993-5:2001, PN-EN 993-5:2001 and strength tests were carried out according to EN 993-5:2018 [20].

Research methodology

Compressive strength testing was carried out on 12 cylindrical specimens with a height of 50 mm and a base diameter of Φ50 mm (±0.5 mm) – 6 specimens cut from a block formed at atmospheric pressure and 6 specimens cut from a block formed at reduced pressure. Three samples from each block were burned out at 1600°C. The samples were dried and stored in a desiccator before testing.

The samples were cut according to the moulding direction. When cutting out, special care was taken to ensure that the surfaces of the cylinder bases were parallel and smooth.

After cutting out, the samples were dried in a laboratory dryer at 110°C (±5°C) to a constant weight, 3 samples from each block were set aside and the rest were then put into a desiccator and cooled to ambient temperature in the desiccator.

After 3 samples were set aside beforehand, they were burned out at 1600°C in a laboratory oven according to the heating scheme:

- Heating to 1200°C for 4 hours.
- Heating to 1550°C for 2 hours and 55 minutes.
- Heating to 1600°C for 50 minutes and holding for 5 hours.
- Cooling to 100°C for 5 hours.

After sample preparation, the samples were described as STD1, STD2, STD3 and VAC1, VAC2, VAC3 for dried samples and STD1 1600, STD2 1600, STD3 1600 and VAC1 1600, VAC2 1600, VAC3 1600 for fired samples. Prior to testing, height measurements were taken at 4 sample points and 2 diameter measurements were taken. An average was taken from the measurements.

The test was carried out on a MATEST type test press with a range of 2000 kN, at a test speed of 1,0 MPa/s ± 0,1 MPa/s.

Analysis of the results

The results obtained are shown in TABLE 2 and then visualised in the graphs in Fig. 16.

The strength of refractory materials is directly proportional to their porosity. This is a relationship generally known for most materials [4,5]. It is also confirmed for the refractories analysed in this study. A large impact of the reduction of porosity on the increase of strength (an increase of 49% for fired samples and 24% for dried samples)

TABLE 2

Summary of compressive strength results.

Samples dried at 300°C					
	Nr	**Diameter**	**Height**	**Compressive strength**	**Average**
		[mm]	**[mm]**	**[MPa]**	**[MPa]**
STD	1	49,54	49,76	94,69	93,42
	2	49,64	49,95	91,95	
	3	49,28	49,55	93,63	
VAC	1	49,36	49,8	115,76	119,53
	2	49,21	48,91	131,11	
	3	49,42	50,46	111,72	
Samples burned out at 1600°C					
STD	1	50,4	50,56	114,52	126,84
	2	49,79	50,35	147,57	
	3	49,99	50,84	118,44	
VAC	1	50,38	50,47	137,84	158,96
	2	50,6	50,06	173,79	
	3	50,39	50,27	165,24	

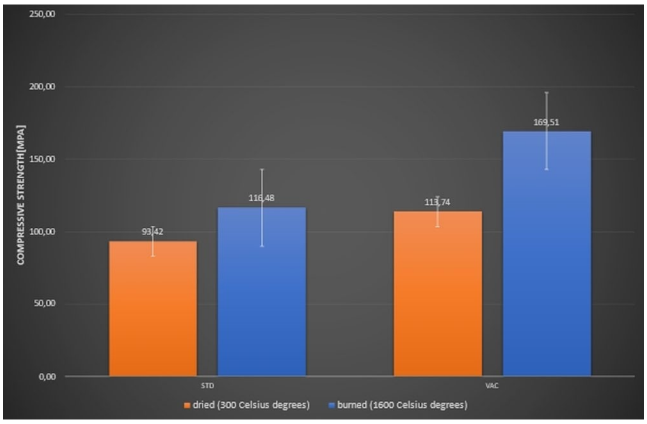


Fig. 16. Summary of compressive strength test results for analysed specimens

7. Summary

The use of a reduced-pressure atmosphere in the moulding of refractories is an innovative solution. This has made it possible to obtain a product with several times less porosity compared to a product formed using traditional methods. Thus, at a relatively low cost and with little complexity of the vacuum installation, it is possible to improve the parameters responsible for the quality of the final product. The results obtained and their analysis from experience show a significant increase in the compressive strength values of the refractory, a more homogeneous structure and smaller and less visible pores. The surfaces are more homogenous, compact, which will allow less degradation (corrosion) of the material during operation in the furnaces extending their service life. The end result is also an increase of cost-effectiveness of metal smelting, heat treatment, etc.

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