Development ceramic floor tiles with increased shear and pressure strengths

LÁSZLÓ A. GÖMZE ▪ Institute of Ceramics and Polymer Engineering, University of Miskolc ▪ femgomze@uni-miskolc.hu

SERGEI N. KULKOV ▪ Institute of Strength Physics and Materials Science SB RAS, National Research Tomsk State University ▪ kulkov@ms.tsc.ru

EMESE KUROVICS ▪ Institute of Ceramics and Polymer Engineering, University of Miskolc ▪ fememese@uni-miskolc.hu

ALES S. BUYAKOV ▪ Institute of Strength Physics and Materials Science SB RAS, National Research Tomsk State University ▪ alesbuyakov@gmail.com

ALEXANDR Y. BUZIMOV ▪ Institute of Strength Physics and Materials Science SB RAS, National Research Tomsk State University ▪ buzimov92@gmail.com

MIHAIL V. GRIGORIEV ▪ Institute of Strength Physics and Materials Science of RAS, Tomsk Polytechnic University ▪ grv@ispms.ru

BRONISLAV I. KANEV ▪ Institute of Ceramics and Polymer Engineering, University of Miskolc ▪ slava.kanev.1995@mail.ru

TATIANA V. KOLMAKOVA ▪ Institute of Strength Physics and Materials Science SB RAS, Tomsk State University ▪ kolmakova@ftf.tsu.ru

RUSLAN V. LEVKOV ▪ Institute of Strength Physics and Materials Science of Siberian Branch of Russian Academy of Sciences, National Research Tomsk Polytechnic University ▪ levkov.r.v@mail.ru

SERGEY A. SITKEVICH ▪ Institute of Ceramics and Polymer Engineering, University of Miskolc ▪ sitx0113@yandex.ru

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Abstract

In the present research work is studied and described the influence of alumina and zirconia powders on physical and mechanical properties of the conventional ceramic floor tiles. After testing several different raw material compositions the authors have found that adding 5 m% commercial ZrO_2 powders the shear strengths and pressure strengths of these floor tiles can be considerable increased. These new floor tiles with increased mechanical strengths may be suitable for use in dairy, meat, food and other industries.

Keywords: alumina, ceramics, compacting, density, floor tiles, mixtures, powders, zirconia

1. Introduction

The ceramic floor tiles are widely used as building materials both for interior architectural purposes in the living and public buildings and for hygiene purposes in the dairy, meat, food and other industries [1-2]. Because of this circumstance there are several research works which are studying, analyzing and describing the physical, chemical, thermal, acoustic and morphological properties both of the used raw materials and the sintered glazed or unglazed floor tiles [3-12]. Generally the production lines of wall and floor tiles industry are used fast drying and sintering technology with roller furnaces of less than 50 minutes cold to cold firing cycle. Because of this fast heating and cooling the green bodies of tiles are very sensitive to the diffusion of water vapor and heating curve in the beginning part of the fast firing roller kilns.

The beautiful designed wall and floor tiles which are excellent in the living and public buildings usually cannot be used in the industrial workshops, storage pools, silos and reservoirs because of the shortages of their durability and mechanical strengths. It can be possible to increase the mechanical strengths through increment of sintering temperature [13] but this is not so easy because of the low melting temperature of

some of the components of the used pulverized powder-mix raw materials. Generally it is possible to get ceramic products with increased mechanical strengths through composite material structures [14-18]. Our aims are to increase the mechanical properties and durability of the ceramic floor tiles used in food and chemical industries by creating a new ceramic composite structure adding ceramic powders with increased mechanical

László A. GÖMZE,

Establisher (in July 1st, 1999) of the Department of Ceramics and Silicate Engineering in the University of Miskolc, Hungary. He is author or coauthor of 2 patents, 6 books and about 300 scientific papers. Recently, he is the chair of the International Organization Board of ic-cmp5 the 5th International Conference on Competitive Materials and Technological Processes (2018) and ic-rmm3 the 3rd International Conference on Rheology and Modeling of Materials (2017)

Sergei N. KULKOV,

is head of Department of Ceramics in the Institute of Strength Physics and Materials Science of the Russian Academy of Science Author of 5 books, more than 150 articles and 18 patents.

Emese KUROVICS,

Graduated in the University of Miskolc, Department of Ceramics and Silicate Engineering as a material engineer, where she actually continues her study as PhD student under supervision of Prof. L. A. Gömze.

Ales S. BUYAKOV,

is a master student at Tomsk State University. He has several reports on international conferences including Junior EuroMAT-2014.

Alexandr Y. BUZIMOV,

PhD student in the Institute of Strength Physics and Materials Science of the Russian Academy of Sciences in Tomsk

Mihail V. GRIGORIEV,

(2014) PhD has finished the Polytechnical Institute of the Siberian Federal University in 2007. At present time he is a post-doctoral fellow in Institute of Strength Physics and Materials Science of Siberian Branch of the Russian Academy of Science under guidance Prof. Kulkov. Topic of his PhD. work was „Features of deformation and fracture of porous alumina ceramics based on nanopowders”

Bronislav I. KANEV,

is graduated master's courses at the Syktyvkar State University named after P. Sorokin in the specialty Geology. At present he is a PhD student at University of Miskolc under supervision of Prof. L. A. Gömze.

Tatiana V. KOLMAKOVA,

lecturer PhD in the Tomsk State University

Ruslan V. LEVKOV

is graduated in the National Research Tomsk State University. At present he is working in the Institute of Strength Physics and Materials Science SB RAS.

Sergey A. SITKEVICH,

is graduated master's courses at the Syktyvkar State University named after P. Sorokin in the specialty Geology. At present he is a PhD student at University of Miskolc under supervision of Prof. L. A. Gömze.

strengths, hardness and durability to the conventional raw materials as pulverized floor tile powders.

2. Materials and experiments

In the last 10-15 years ceramics and ceramic matrix composite materials (CMC) are widely used because of their low density and excellent wear resistance, hardness, toughness and mechanical strength [19-24]. Really the most widely used and popular technical ceramics [25-33] have relatively low density and very competitive mechanical properties (Table 1).

Type of ceramics	Density g/cm ³	Tensile strength MPa	Bending strength MPa	Compressive strength MPa	Young Modulus GPa	Fracture toughness MPam ^{1/2}
Al ₂ O ₃	3.98	210	560	2800	392	5.5
SiC sintered	3.1	175	560	3920	420	4.4
Si ₃ N ₄ reactive bound	2.5	140	245	1050	210	3.3
Si ₃ N ₄ hot pressed	3.2	560	910	3500	315	5.5
SiAlON	3.24	420	980	3500	315	9.9
ZrO ₂ partially stabilized zirconia (PSZ)	5.8	455	700	1890	210	11.0
ZrO ₂ transformation toughened zirconia (TTZ)	5.8	350	806	1750	203	12.1

Table 1. The mechanical properties of the most widely used technical ceramics
1. táblázat A leggyakrabban alkalmazott műszaki kerámiák mechanikai tulajdonságai

In the laboratory experiments were used conventional pulverized powder mixtures of floor tiles which were prepared from the following raw materials: clay Petény I and II, clay Teplicsány, rhyolite, conventional clay Kisőrs, GVZ and phonolite. The mineralogical composition of these powder mixtures is described in the Table 2 and the typical microstructure and chemical components are shown in Figure 1.

Minerals	Chemical composition	Quantity, m%
β-quartz	SiO ₂	22.51
α-quartz	SiO ₂	37.31
tridimite	SiO ₂	7.35
orthoclase	K(Al, Fe)Si ₂ O ₈	0.3
nepheline	3Na ₂ O K ₂ O 4Al ₂ O ₃ 9 SiO ₂	5.37
albite	NaAl ₃ Si ₃ O ₁₁	4.55
microcline	K Al ₃ Si ₃ O ₁₁	5
illite	2K ₂ O 3MgO Al ₂ O ₃ 24 SiO ₂ 12H ₂ O	3.69
sanidine	(Na, K)(Si ₃ Al)O ₈	2.89
glauconit-1	K(Fe, Al) ₂ (Si, Al) ₄ O ₁₀ (OH) ₂	3.32
sodium aluminum silicate	Na ₂ O SiO ₂ Al ₂ O ₃	6.83
sodium iron oxid	Na ₄ FeO ₃	0.93

Table 2. The mineralogical composition of the used floor tile powder mixtures
2. táblázat A hagyományos padlólap porkeverék ásványi összetétele

Fig. 1. Micrograph and chemical composition of the conventional pulverized powder of traditional floor tiles

1. ábra A hagyományos padlólap atomizor por mikroszerkezete és kémiai összetétele

On the basis of the earlier experiments with alumina [34-37] and zirconia [38-40] of the authors in this research to the conventional floor tiles powder Martoxid KMS-94 alumina (Figure 2) and TZP zircon-dioxide (Figure 3) powders were added in different ratios.

Fig. 2. Micrograph and chemical composition of the Martoxid KMS-94 alumina

2. ábra Martoxid KMS-94 aluminium-oxid mikroszerkezete és kémiai összetétele

Fig. 3. Micrograph and chemical composition of the TZP zircon-dioxide

3. ábra TZP cirkon-dioxid mikroszerkezete és kémiai összetétele

To increase the mechanical properties to the conventional pulverized floor tiles powders Al₂O₃ (A) and commercial ZrO₂ (Z) powders were added in portion of 5m%, 10m%, 15m% and 20m% as they are shown in Table 3.

Mixture sign	Pulverized powder	Martoxid KMS-94 alumina	TZP zircon-dioxide
A5	95	5	-
A10	90	10	-
A15	85	15	-
A20	80	20	-
Z5	95	-	5
Z10	90	-	10
Z15	85	-	15
Z20	80	-	20
ZA5	95	2.5	2.5
ZA10	90	5	5
ZA15	85	7.5	7.5
ZA20	80	10	10

Table 3. The composition of prepared new mixtures by m%
3. táblázat A készített keverékek összetétele tömegszázalékban [m%]

From each mixture of the powders were taken out by 200g weights and milled and mixed in a Retch PM 400 planetary ball mill at 200rpm trough 5, 10 and 15 minutes. The so prepared powder mixtures were divided by 10g and compacted through one-sided pressing to discs in a cylindrical die cavity with 20mm diameter and filling depth of 50mm on uniaxial mechanical press at compressing pressures of 158MPa, 196MPa, 234MPa, 274MPa and 312MPa. After compacting the geometrical parameters of the discs were measured and determined the “green” density of the specimens. Further these discs were sintered in an electrical laboratory kiln heating up to 1250°C. When the kiln achieved this temperature the current was switched off and the kiln cooled down freely. The influence of the material composition on the color of the sintered specimens is very well seen in the Figure 4.

Fig. 4. The sintered at 1250°C ceramic disc specimens with different volumes of Al_2O_3 and ZrO_2 powders in the raw material mixtures

4. ábra A különböző mennyiségű Al_2O_3 és ZrO_2 port tartalmazó keverékekből préselt, majd szinterelt próbatestek

3. Result and discussions

During the experiments the green density of the pressed specimens were determined depending on the quantity (portion) of the zirconia and on milling times as function of the compaction pressure. It is obvious from Table 1 that density of zirconia is 5.8 g/cm³ which is much higher than the density of conventional floor tiles after sintering. So with increasing the portion of TZP powders in the mixtures will increase the green density of the pressed specimens. At the same time the densities of the pressed specimens are increased also with increments the values of compression pressures (Figure 5).

Fig. 5. Influence of volume of ZrO_2 powders and compaction pressure on the density of the pressed “green” specimens

5. ábra A ZrO_2 por mennyiségének és a présnyomás hatása a préselt nyers próbatest sűrűségére

The influence of milling times also was studied at different compaction pressures (Figure 6). For these 2 different mixtures

were prepared with 5m% of zirconia (a) and 5m% of alumina (b) and milled and mixed in a Retch PM 400 planetary ball mill at 200rpm trough 5, 10 and 15 minutes. In both cases there is no remarkable different between the pressed green densities after 5 min and 10 min milling but after 15 minutes of milling the densities of the pressed green specimens have decreased considerable. This phenomenon can be explained with intensive mechanochemical processes taking place during fine comminution of the ceramic powders. Thanking to this mechanochemical processes the powder mixtures have very fine grain and pore structures and the submicron particles are electro statically repel each others in the compacting die cavity during and after pressing.

Fig. 6. Influence of the milling time on density of the pressed specimens depending on compaction pressure

6. ábra Az őrlési idő hatása a préselt nyers próbatest sűrűségére az alkalmazott présnyomás függvényében

While the green densities of the pressed specimens are followed the densities of the used high-tech ceramics powders after sintering the specimens with TZP zirconia additives had the smallest densities, meanwhile the $\text{ZrO}_2 + \text{Al}_2\text{O}_3$ mixed additives have generated the highest sintered densities (Figure 7).

Fig. 7. Influence of volume of Al_2O_3 and ZrO_2 additive powders on density of the sintered specimens

7. ábra Az Al_2O_3 és ZrO_2 por adalékanyag mennyiségének a hatása a szinterelt próbatestek sűrűségére

After the sintering the cylindrical disc specimens were tested on shear strength and pressure strength depending on the containments of zirconia (ZrO_2) and alumina (Al_2O_3) powders in the raw material mixtures. The typical destruction and fracture surfaces of the cracked specimens are shown in Figure 8 and the average values of the shear strengths and pressure strengths are shown in Figure 9.

Fig. 8. Typical fracture surfaces of specimens after shear test (a) and pressure test (b)
8. ábra A próbatestek tipikus törés felületei a nyíró- (a) és a nyomószilárdság vizsgálatakor (b) után

It is obvious from the Figure 9 that the volumes of Al_2O_3 and ZrO_2 raw materials very strong influence both on the shear strengths (a) and pressure strengths (b) of the floor tile specimens sintered at $1250^\circ C$. The highest values of shear strengths were achieved when 5m% zirconia powder was mixed into raw materials and the smallest when 15m% of ($Al_2O_3 + ZrO_2$) mixed powders were added. The pressure strengths are also minimum when 15m% of Al_2O_3 itself and 15m% of ($Al_2O_3 + ZrO_2$) additives are mixed into the conventional floor tile pulverized powders.

Fig. 9. The shear strengths (a) and pressure strengths (b) of the sintered specimens made from new raw material compositions

9. ábra A szinterelt próbatestek nyírószilárdsága (a) és nyomószilárdsága (b)

In cases when only alumina powder additives are used the shear strengths of the sintered specimens are higher in each case than their pressure strengths at 10m%, 15m% and 20m%. This phenomenon can be explained that the used $1250^\circ C$ sintering temperature is not high enough when the volume of Al_2O_3 is higher than 5 m%. At the same time the largest pressure strengths of the floor tile specimens made from the new powder mixtures were achieved at volume of 20m% ZrO_2 but at 5m% ZrO_2 also very good pressure strengths were achieved with average values about 45MPa.

4. Conclusion

It is obvious from the realizes experiments that the traditional raw material composition can be used to produce ceramic floor tiles with increased mechanical shear and pressure strengths just adding to its 5m% commercial ZrO_2 powders and sintering it at $1250^\circ C$.

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Növelt nyíró- és nyomószilárdságú kerámia padlólapok fejlesztése

Jelen kutatás során tanulmányozták és leírták az alumínium-oxid és a cirkon-dioxid hatását a hagyományos kerámia padlólapok fizikai és mechanikai tulajdonságaira. Különböző nyersanyag összetételek vizsgálata után a szerzők megállapították, hogy a kereskedelmi forgalomban levő ZrO₂ por 5 m%-ban történő hozzáadásával jelentősen növelhető a padlólapok nyíró- és nyomószilárdsága. Az ilyen új, megnövelt mechanikai szilárdsággal rendelkező padlólapok alkalmasak lehetnek a tej-, hús-, élelmiszer- és más iparágakban történő felhasználásra.

Kulcsszavak: alumínium-oxid, kerámiák, préselés, sűrűség, padlólapok, keverékek, porok, cirkon-dioxid

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