

Synthesis of Micro- and Mesoporous SiO₂ Based Ceramic Materials (Using Sol-gel Technology)

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Abstract – The aim of research is to develop an area of micro- and mesoporous ceramic materials of ternary - TiO₂-ZrO₂-SiO₂ system. Ceramic system TiO₂-ZrO₂-SiO₂ has been synthesized via a sol-gel technology by hydrolysis of tetraethylorthosilicate (TEOS) and zirconia propoxide and titanium isopropoxide solutions. The sols have been polymerized at room temperature to obtain gels and dried at 100 °C for 72 hours. Xerogel has been milled for 1 or 6 hours and axially pressed for nanoporous ceramic samples. Powders have been sintered at 800 °C or 1000 °C in air. 1 M hydrochloric acid and distilled water have been used as a catalyst to advance the hydrolysis and condensation reactions. All samples have been characterized by X-ray (XRD) diffraction, particle size distribution, crystallite size distribution, compressive strength. Porosity and specific surface area of nanoporous ceramic samples have been determined by Brunauer, Emmett and Teller (BET) nitrogen adsorption-desorption isotherms.

Keywords – Nanoporous ceramic materials, sol-gel technology, TiO₂-ZrO₂-SiO₂.

I. INTRODUCTION

The properties of silica dioxide (SiO₂) have been extensively studied by many researchers due to its wide range of applications.

There are many publications about SiO₂ that can be used as basic (standard) oxide. Researchers have published numerous variations of the synthesis conditions (e.g., types of technologies, thermal treatment, compositions) which cause modification in the structure of dense or porous ceramic material based on SiO₂. The traditional silica-containing materials were prepared using tetraethoxysilane (TEOS), fumed silica, or water glass as silica resources. Recently, attention has been paid to a new process to form mesoporous silica from natural clays and other layered silicate materials through hydroleaching processes [1]. There is a growing interest in the synthesis of nanoporous binary or ternary oxide materials containing silica dioxide. The investigation of the structures that can be used for adsorption, catalysis or detection of molecules is very urgent.

As it is known, pure metal dioxides such as TiO₂ and ZrO₂ have a very small specific surface area, low thermal stability and high price, which make them unsuitable for industrial applications. Thus, the development of mixed oxide systems attracts increasing attention. The homogeneous incorporation of Ti and Zr into a SiO₂ matrix is important to obtain materials that exhibit the chemical, thermal and mechanical stability [2].

Zirconium titanate (ZrTiO₄) is an intermediate compound in the binary system ZrO₂-TiO₂. ZrTiO₄ is a well-known compound in the field of electroceramics, where it has been used in dielectric resonators and materials for

telecommunications [3], in dielectric resonators for microwave telecommunications [4], but in most cases as a dense ceramic material. Nanoporous materials are of the groups of nanomaterials. Their typical structure of pores in nanometer dimensions makes them useful for various applications. As it is known, according to the International Union of Pure and Applied Chemistry (IUPAC), porous materials can be classified into three groups: (1) micropores (smaller than 2 nm in diameter); (2) mesopores (between 2 nm to 50 nm in diameter); (3) macropores (larger than 50 nm in diameter).

Materials with tailor-made pore size distribution, open or closed porosity and shapes are particularly important in applications, where molecular recognition is necessary, such as: shape-selective catalysis, selective adsorption, petroleum catalytic cracking and so on [1].

The control of nanoporous structures, mechanical properties, density of nanoporous ceramics are very important factors from the viewpoint of their application. The usual route for nanoporous ceramic preparation is the sol-gel technique. Sol-gel technology is one of the most suitable technologies to prepare ternary complex oxide systems with an atomic level of the ingredients.

The aims of the research are to prepare TiO₂-ZrO₂-SiO₂ system ceramics via the sol-gel technology and to determine microstructural, mechanical properties and porosity of nanoporous ceramics.

II. MATERIALS AND METHODS

In the synthesis procedure of micro- and mesoporous ceramics, the sol was synthesized using tetraethylorthosilicate (TEOS, Aldrich, 99.9 %) as the SiO₂ source, zirconium propoxide (70 wt% solution in propan-1-ol, Aldrich) and titanium isopropoxide (Aldrich) alcocide solution as ZrO₂ and TiO₂ source, propanol-2 (Sigma-Aldrich) was used as a reciprocal solvent [5]. Acetic acid (Sigma – Aldrich), 1M hydrochloric acid (P.P.H.™Stanlab™) and distilled water were used as a catalyst to advance the hydrolysis and condensation reactions. According to the studies [6], a surface area can be varied by the concentration of added hydrochloric acid [6] that exerts influence on morphology, mechanical properties, pore structure and volume of final nanoporous material. Molar percentage ratio of elements TiO₂/ZrO₂/SiO₂ was 10/20/70, 15/15/70 and 20/10/70, but 1 h or 6 h – milling time of powder heat-treated at 500 °C and the heating rate was fixed at 5 °C/min. In all ceramic systems, preparation of sols was a considerable ratio of chemical solutions: alcocide/propanol-2 = 1/8, alcocide/acetic acid = 1/3 and 1 M hydrochloric acid/alcocide = 1/5, TEOS/distilled water = 1/4. Sols were prepared following the steps above, Fig. 1.

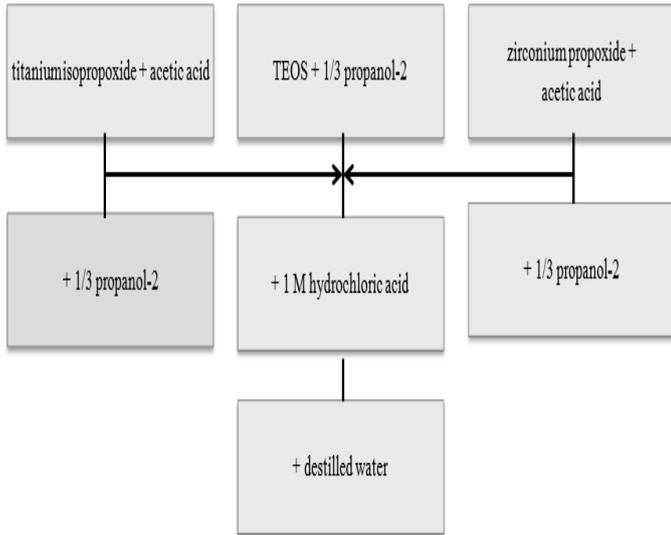


Fig. 1. Preparation of sol.

When the gel was formed, it was dried at 100 °C for 72 h. The xerogel was calcined at 500 °C for 1 hour and the heating rate was fixed at 5°C/min. The obtained powder was milled (planetary ball mill, "Retsch PM 400") for 1 or 6 hours for particle homogenization. The speed of ball rotation was 200 rpm. Size distribution of powder particles was determined by "MAS ZetaPALS Brookhaven Instr." using ethyl lactate as surfactant and ethyl alcohol. Ceramic samples were axially pressed by manual press (SPRUT 10/185, Latvia), pressure 220 bars, time 20 seconds. For the compressive strength ("Compression Test Plant Toni Norm, Toni Technik by Zwick", ultimate 300 kN, program "Setsoft 2000") tests, the nanoporous ceramic samples were sintered at 800 °C or 1000 °C for 1 hour and the heating rate was fixed at 5 °C/min. Ceramic samples – cylinders, whose diameter was 1.2 cm, but height was more than 1.2 cm. All the samples were prepared following a scheme that is shown in Fig. 2.

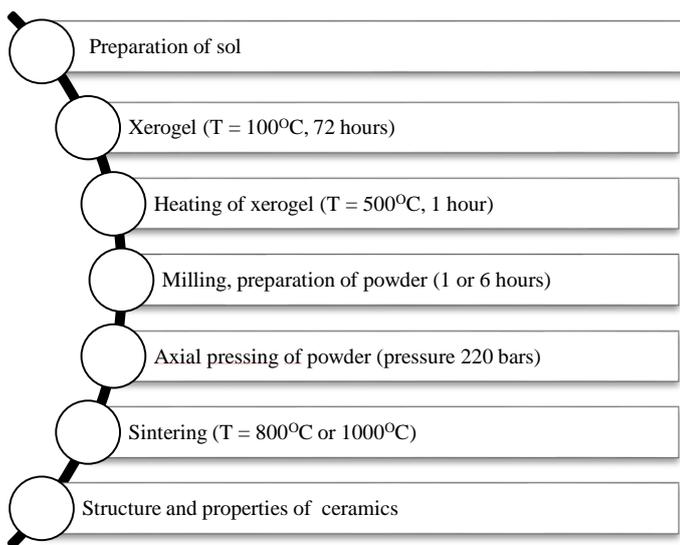


Fig. 2. Preparation of micro- and mesoporous ceramic samples.

The quality of samples was characterized by X-ray (XRD) diffraction data collected using a Rigaku Ultima+ (Japan) diffractometer. Cu K_α wavelength was used, the scan conditions were 2^θ, but 2 Θ mode – for the range over which the diffraction patterns were recorded. The crystallite size was evaluated using the Debaj – Schreder equation:

$$D = \frac{\lambda}{B \cdot \cos\theta} \quad (1)$$

where *D* – is the crystallite size, nm;
 λ – the length of x-radiation wave, nm;
B – the full width at half maximum of the diffraction peak;
 θ – the Wulf – Breg angle.

Apparent density of nanoporous ceramic samples was evaluated using the following equation:

$$\rho_{ap} = \frac{g_o \times \rho_{H_2O}}{(g_1 - g_2)} \quad (2)$$

where ρ_{ap} – apparent density, g/cm³;
g_o – mass of dry ceramic sample, g;
g₁ – mass of watered ceramic sample into air, g;
g₂ – mass of watered ceramic sample into water, g;
 ρ_{H_2O} – water density, g/cm³.

The specific surface areas of all ceramic samples were determined by Brunauer, Emmett and Teller (BET) nitrogen adsorption-desorption isotherms and were recorded by using "Nova 1200 E-Series, Quantachrome Instruments" (for pore size of 0.35 nm – 200 nm). Scanning electron microscope (SEM) "Hitachi Table Top Microscope TM3000" was used for the analysis of morphology.

III. RESULTS AND DISCUSSION

It is known that physical properties and quality of ceramic powder are affected by the morphology, particle size distribution and chemical composition. As it is known, high-energy ball – milling of TiO₂-ZrO₂-SiO₂ system powders promoted changes in particle size. Changes in phase transitions and particle size decrease depended on powder-ball weight ratio, milling time and speed. Figure 3 summarizes the nanoscale powder size distribution of all ceramic series after 1- or 6-hour milling time. After 6 hours, milling samples exhibited wider particle size distribution (shown as dispersion), but their average particle size (shown as a column) decreased. It was noticed that the grain size of the as-milled powders had already been reduced to tens of nanometers [7], and it could be the main reason of a decrease in powder particle size. As shown, larger particles were in composition with higher mol% TiO₂ in the structure. After 6 hours, milling samples exhibited the decrease in an average particle size by 20.8 % to 72.9 %.

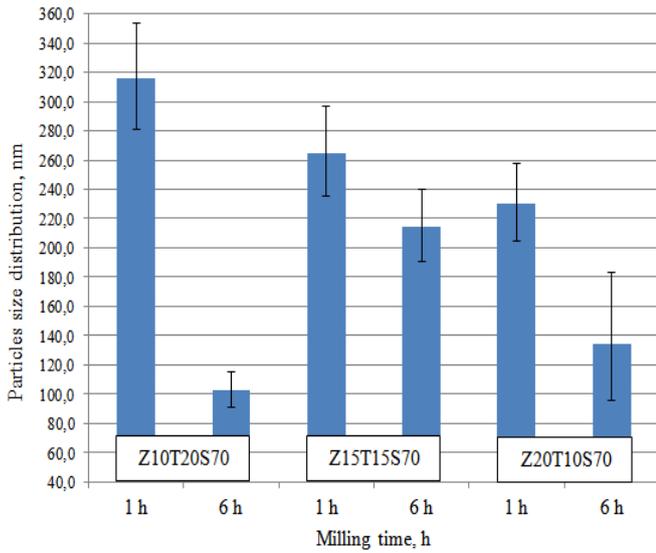
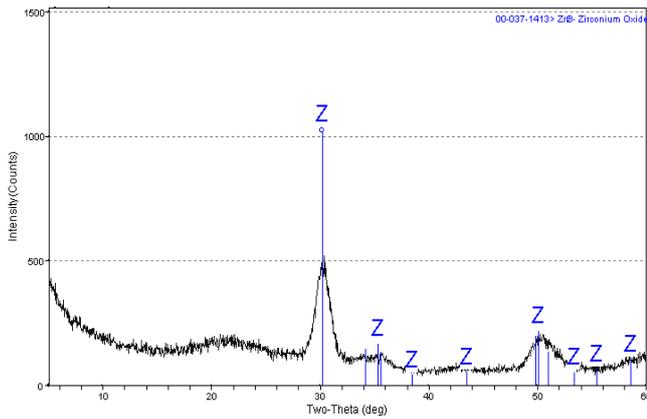
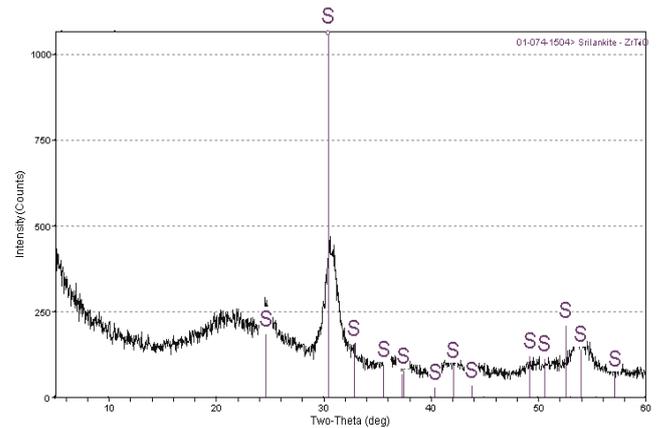


Fig. 3. Particle size distribution of powders after 1- or 6-hour milling time.

X-ray diffraction patterns showed that milling time necessary to cause the phase transition in powder samples of TiO₂-ZrO₂-SiO₂ system depended on milling time and composition of sol. Figure 4 (a, b) and Figure 5 (a, b) show the results of X-ray diffraction patterns for two series of powder samples (molar ratio of ZrO₂/TiO₂/SiO₂: a) 20/10/70, b) 10/20/70) heat-treated for 1 hour at 1000 °C, but milling time was different: 1 hour (Fig. 4 a, b) and 6 hours (Fig. 5 a, b). XRD patterns exhibited the peaks of the monoclinic zirconia phase if powder was milled for 1 hour and contained 20 mol% of ZrO₂ into primary sol. If primary sol contained less than 20 mol% ZrO₂ XRD patterns exhibited the peaks of zirconium titanate (ZrTiO₄, srilankite). Sol-gel technology is based on the hydrolysis and condensation reaction of alcoxide solutions. As it is known, the reaction time of different alcoxides is not the same. The silicon atoms carry substantially less positive charge; thus, the hydrolysis and condensation reaction of silicon alcoxides occur at much lower rates [8]. This order corresponds to the reactivity sequence of tetravalent alcoxides in hydrolysis reactions: Si(OiPr)₄ << Ti(OiPr)₄ < Zr(OiPr)₄ [9]. It is supposed that phase formation is caused by the used molar % alcoxides into primary sol.

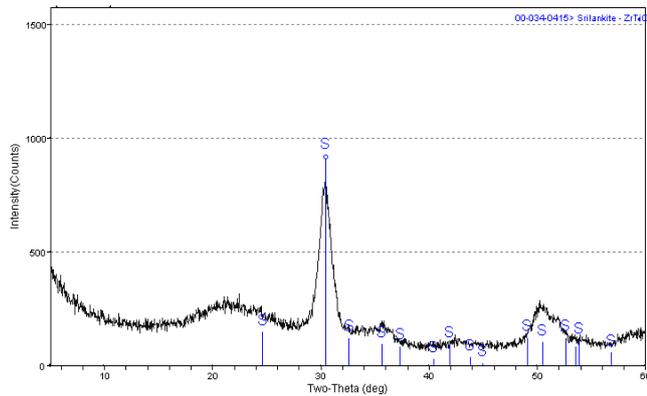


a)

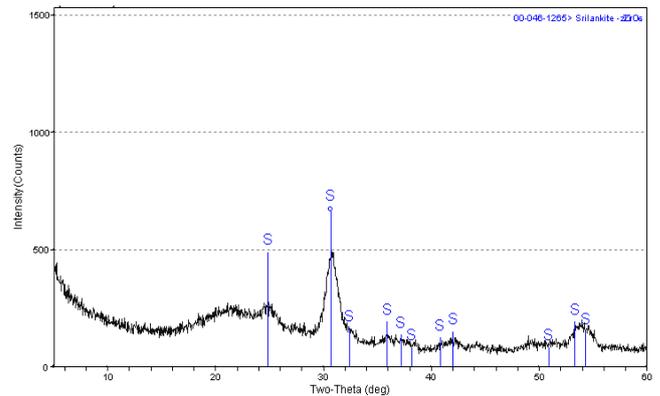


b)

Fig. 4. XRD – milling time was 1 hour, 1000 °C, molar ratio of ZrO₂/TiO₂/SiO₂: a) 20/10/70, b) 10/20/70.



a)



b)

Fig. 5. XRD – milling time of powder was 6 hours, sintering temperature was 1000 °C, mol% ratio of ZrO₂/TiO₂/SiO₂: a) 20/10/70, b) 10/20/70.

TABLE 1
XRD PATTERNS OF MICRO- AND MESOPOROUS CERAMICS, SAMPLES SINTERED AT 1000 °C

Powder milling time, hours	Molar ratio of ZrO ₂ -TiO ₂ -SiO ₂	Reference code	Chemical formula	Compound name	Mineral	Crystal system	Space group
1	10/20/70	01-074-1504	ZrTiO ₄	Zirconium titanium oxide	Srilankite	Orthorhombic	Pnab
	15/15/70	01-074-1504	ZrTiO ₄	Zirconium titanium oxide	Srilankite	Orthorhombic	Pnab
	20/15/70	00-037-1413	ZrO ₂	Zirconium oxide		Orthorhombic	Pbma
6	10/20/70	00-046-1265	Ti ₂ ZrO ₆	Zirconium titanium oxide	Srilankite	Orthorhombic	Pnab
	15/15/70	00-046-1265	Ti ₂ ZrO ₆	Zirconium titanium oxide	Srilankite	Orthorhombic	Pnab
	20/15/70	00-034-0415	ZrTiO ₄	Zirconium titanium oxide	Srilankite	Orthorhombic	Pnab

For the samples milled for 6 hours, XRD patterns are shown in Fig. 4 (b) and 5 (b), and Table 1 demonstrates the phase transition from ZrO₂ to zirconium titanate oxide (ZrTiO₄) as well as from ZrTiO₄ to Ti₂ZrO₆. The analytical results are based on the identification using Rigaku Ultima+ (Japan), and the database results are summarized in Table 1. The traditional preparation of TiZrO₄ ceramics was based on solid state reactions between TiO₂ and ZrO₂ powders at high temperatures (above 1400 °C) [5]. This fact could be the main reason for phase transitions. Using sol-gel technology it is possible to produce TiZrO₄ ceramics at lower temperatures (1000 °C).

Figure 6 summarizes the crystallite size distribution of meso- and macroporous ceramic samples. Average crystallite size was determined based on the broadening of the XRD maxima by Scherrer equation, using ZrO₂, ZrTiO₄ and Ti₂ZrO₆ samples sintered at 800 °C and 1000 °C for 1 and 6 hours of milling time. The peak intensity of X-ray diffraction (XRD) patterns for powder samples (for ZrO₂, ZrTiO₄ and Ti₂ZrO₆) increases with the sintering temperatures indicating the crystal growth of all crystalline phases. The diagram shows that the milling time exerts an effect on crystallite size distribution – a longer period of high-energy milling reduces the crystallite size. However, higher physical agglomeration of crystallites originates from higher surface energy of smaller crystallites (sample Z15T15S70, milling time of 6 hours). The crystallite size sintered at 1000 °C is from 48.6 nm to 106.8 nm and testifies the formation of a more arranged structure. The increase in a sintering temperature promotes the crystallite growth. There is a correlation of results between the crystallite size and particle size distribution.

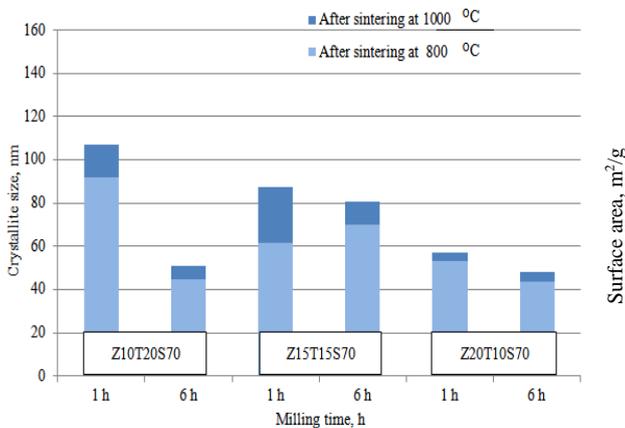


Fig. 6. Crystallite size distribution.

Figure 7 summarizes the results of macro- and mesoporous ceramic samples according to compressing strength tests. The histogram shows that the higher compressive strength is characteristic of samples sintered at 1000 °C of all ceramic series. Compressive strength after sintering at 800 °C is in the range of 38.7 MPa – 54.1 MPa, but after sintering at 1000 °C from 41.5 MPa to 68.2 MPa. Another effect is shown by powder milling time. In case of a longer period of milling time, ceramic samples exhibit better compressive strength in comparison with the ones milled only for one hour. Phase transition and pores into an external wall of ceramic samples substantially influence compressive strength.

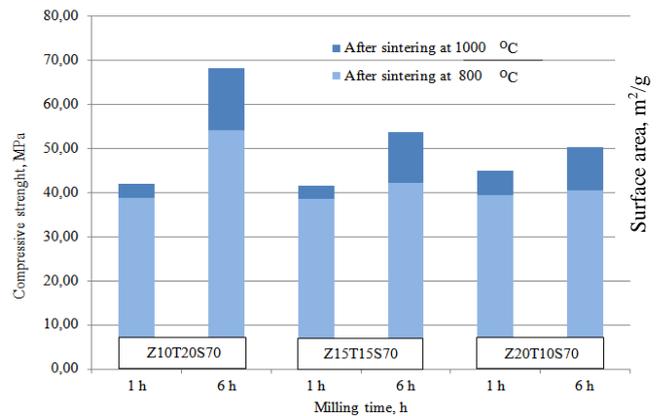


Fig. 7. Compressive strength.

Figure 8 summarizes the results of apparent density of ceramics after sintering at 800 °C and 1000 °C. After sintering at 800 °C, the apparent density of the ceramic samples ranged from 1.83 g/m³ to 1.87 g/m³, but after sintering at 1000 °C it increased in the range of 2.02 g/m³ – 2.31 g/m³. After sintering at 1000 °C, the compressive strength of the samples increased; moreover, changes in volume of nanosize pores and pore size distribution were observed.

The pore size distribution of micro- and mesoporous ceramics was determined using nitrogen adsorption-desorption measurements. Figure 9 (a, b) summarizes the results of surface area histogram of micro- and mesoporous ceramic samples after sintering at 800 °C or 1000 °C, but for a different period of milling time: a) 1 hour, b) 6 hours. BET results show that a larger surface area is characteristic of ceramic samples after sintering at 800 °C, but it decreases essentially after sintering at 1000 °C. A larger surface area was observed in cases, when half pore width was in the range from ≈ 0.84 nm – 0.92 nm of sample Z15T15S70

(1 hour). After 6 hours of milling time, a larger surface area was observed in cases, when half pore width was in the range from ≈ 0.96 nm – 1.1 nm of sample Z10T20S70.

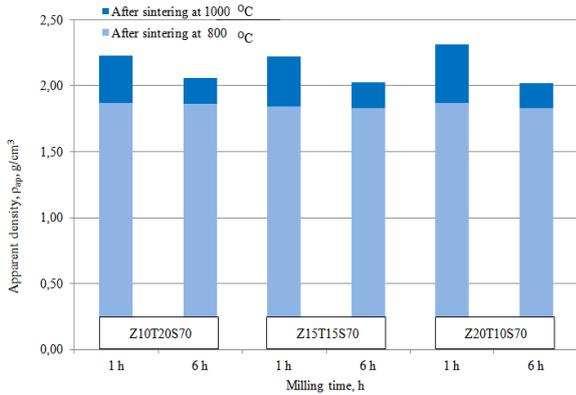
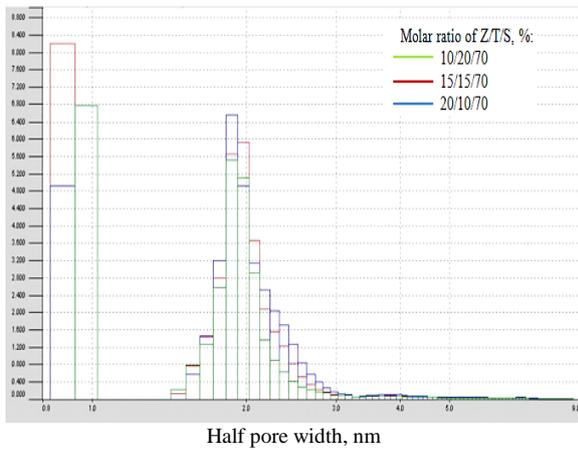


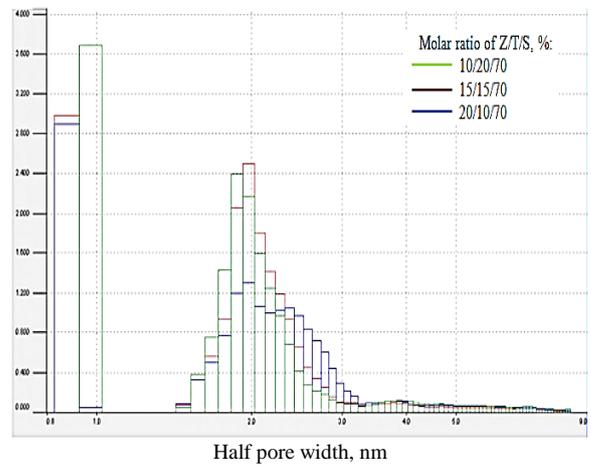
Fig. 8. Apparent density of ceramic samples after sintering at 800 °C or 1000 °C.

The observed surface area values depend on the powder milling time and the different mol% ratio of the ceramic samples. In general, pore volume could be observed by addition of ZrO₂. The surface area of the ternary system TiO₂-ZrO₂-SiO₂ decreases, if volume of ZrO₂ increases.

Figure 10 (a, b) shows SEM images of micro- and mesoporous ceramic samples after sintering at 1000 °C and different milling time. As it can be seen from the SEM images, ceramic samples pressed from powder milled for 1 h have comparatively large particles. In photos taken from ceramic samples pressed from powder milled for 6 h, distribution of particle size is relatively small, but there are seen larger sintered regions. The particle size significantly decreases by increasing the milling time. Pores have become comparatively smaller, and denser areas can be observed that could be formed by sintering particles.

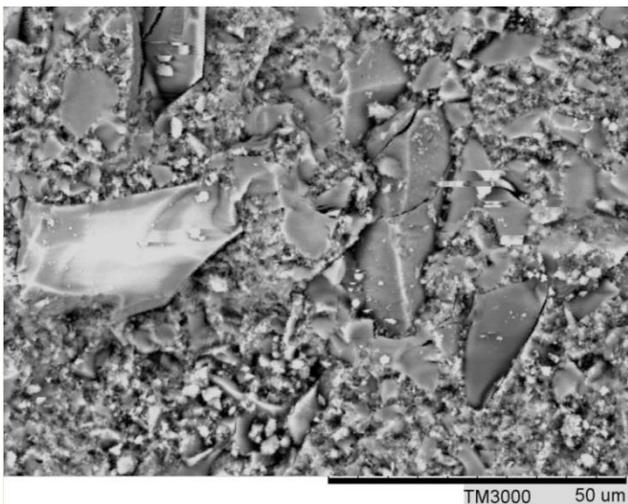


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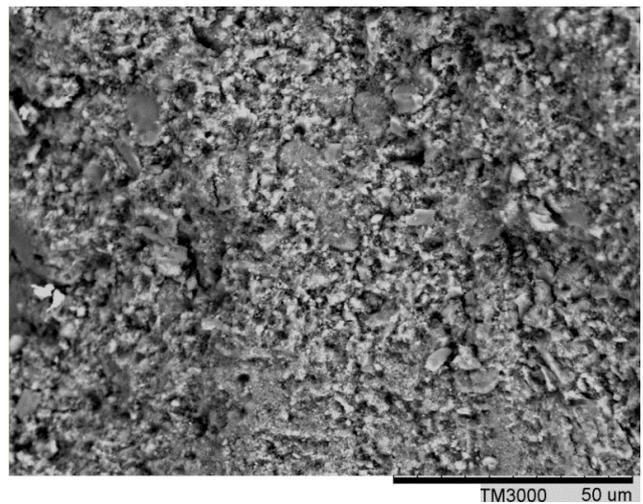


b)

Fig. 9. BET surface area of ceramic samples after sintering at 1000 °C. Samples were pressed from powder milled for different periods of time: a) 1 hour, b) 6 hours.



a)



b)

Fig. 10. SEM images of micro- and mesoporous ceramic Z10T20S70 after sintering at 1000 °C. Samples were pressed from powder milled for different periods of time: a) 1 hour, b) 6 hours

VI. CONCLUSIONS

Micro- and mesoporous ceramics of the system $\text{TiO}_2\text{-ZrO}_2\text{-SiO}_2$ were prepared from silicon, titanium and zirconium alcoxide by sol-gel technology. Content of SiO_2 was 70 mol% of all ceramic sample series. Influence of powder milling time on the structural and mechanical properties was investigated.

The micro- and mesoporous ceramic samples were prepared by sintering at 800 °C and 1000 °C. Powders were milled for 1 or 6 hours. The average particle size after 1 hour-long milling time was in the range of 230.1 nm – 315.7 nm, but after 6 hour-long milling time it was from 102.5 nm – 214.1 nm.

Regardless of molarity of the added alcoxide solution, the phase transition of samples was dependent on the milling time – 1 hour-long milling time leads to the formation of ZrO_2 and ZrTiO_4 , while 6 hours – ZrTiO_4 and Ti_2ZrO_6 . The reason for these transitions could be explained by the solid state reaction caused during the milling process between TiO_2 and ZrO_2 . Essential influence of milling time and volume of metal alcoxide solutions on the apparent density was not observed.

The compressive strength increased essentially if the sample was pressed from powder milled for 6 hours because after sintering at 1000 °C particles formed dense regions.

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Margarita Karpe, Gundars Mežinskis. Mikro- un mezoporainas SiO_2 saturošas keramikas sintēze ar sola – gēla tehnoloģiju

Viena no būtiskām nanomateriālu grupām ir nanoporaini materiāli. Šiem materiāliem pieņemtais raksturīgais poru diametrs atbilst sekojošam sadalījumam: mikroporas (< 2 nm), mezoporas (no 2 nm – 50 nm) un makroporas (> 50 nm). Porainu materiālu pētījumi būtiski palielinājās 20. gs. 90. gados. Tie piesaistīja zinātnieku interesi ar iespējamo plašo pielietojumu dažādās nozarēs, kurās izmanto adsorbcijas, katalīzes, membrānu materiālus, kā arī separācijas un attīrīšanas procesos, kā arī tiem raksturīgajām īpašībām – zemāku blīvumu un lielāku īpatnējās virsmas laukumu. Sākotnēji pētījums galvenokārt izmantoja silīcija dioksīdu (SiO_2), kas nereti tiek uzskatīts kā etalons. Vēlāk, paplašinoties šai pētījumu jomai (atkarībā no sintēzes tehnoloģijas, temperatūras u.c. parametriem), sāka veidot 2 un 3 komponentu sistēmas. Tā rezultātā radās iespēja sintezēt materiālus, kā arī radīt jaunus, ar vēlamajām īpašībām. Pētījumā aplūkota trīskomponentu sistēma $\text{TiO}_2\text{-ZrO}_2\text{-SiO}_2$, kas sintezēta ar sola-gēla tehnoloģiju. Komponentu attiecība sistēmā: SiO_2 – 70 mol%, taču TiO_2 un ZrO_2 mol% attiecība ir mainīga un sastāda attiecīgi 10, 15, vai 20, nosacīti veidojot trīs atšķirīgas paraugu sērijas. Lai raksturotu iegūto mikro- un mezoporas saturošo keramikas materiālu, atkarībā no metālu mol% attiecības, darba gaitā aplūkota atšķirīga pulvera malšanas laika ietekme, veikta rentgenfāzu difrakcijas analīze, noteikta spiedes stiprība, šķietamais blīvums, kā arī porainība, veicot saķepināšanu 800 °C un 1000 °C temperatūrā. No iegūtajiem rezultātiem redzams, ka malšanas procesa laikā notikusi fāzu pāreja (no ZrO_2 uz TiZrO_2 vai Ti_2ZrO_6) visām trim paraugu sērijām. Iegūtā keramikas materiāla šķietamais blīvums ir no 1,8 g/cm³ līdz 2,3 g/cm³, bet spiedes stiprība ir robežās no 38 MPa līdz 68 MPa, atkarībā no komponentu attiecības, pulvera malšanas ilguma un saķepināšanas temperatūras.

Маргарита Карпе, Гундарс Межинскис. Синтез микро- и мезопористой керамики, содержащей SiO_2 , с помощью золь-гель технологии

Один из разделов наноматериалов занимают нанопористые материалы. Для этих материалов характерен определенный диаметр пор, который соответствует следующему разделению: микропоры (< 2 нм), мезопоры (но 2 нм – 50 нм) и макропоры (> 50 нм). В девяностых годах двадцатого столетия значительно увеличилось число исследований, посвященных пористым материалам. Большое внимание к ним обусловлено их возможным применением в разных областях (адсорбция, катализ, применение в мембранных материалах, в процессах сепарации и очистки), а также такими свойствами, как низкая плотность и большая удельная площадь поверхности. В первичных исследованиях главным образом использовали диоксид кремния SiO_2 , который во многих случаях считается эталоном. В дальнейшем, с увеличением исследований в этой области, в зависимости от технологий синтеза, температуры и других параметров, стали использовать 2 и 3 компонентные системы. В результате чего появилась возможность синтезировать материалы, а также создавать новые, с желаемыми свойствами. В исследовании рассмотрена $\text{TiO}_2\text{-ZrO}_2\text{-SiO}_2$ трехкомпонентная система, которая синтезирована с помощью золь-гель технологии. Отношение компонентов в системе: SiO_2 – 70 моль %, в свою очередь, молярное отношение TiO_2 и ZrO_2 разное и соответственно составляет 10 моль %, 15 моль % и 20 моль %, таким образом, образуя три разные серии образцов. Чтобы характеризовать полученную керамику, содержащую микро- и мезопоры, в зависимости от молярного отношения металлов и температуры спекания 800 °C и 1000 °C, в ходе работы было анализировано влияние времени измельчения порошка, произведен рентгенофазовый анализ, определен предел прочности на сжатие, удельная плотность, а также пористость. Из полученных результатов видно, что для всех трех серий образцов в процессе измельчения произошел фазовый переход (из ZrO_2 в TiZrO_2 или Ti_2ZrO_6). В зависимости от отношения компонентов, длительности измельчения порошка и температуры спекания удельная плотность полученной керамики составляет 1,8 г/см³ – 2,3 г/см³, предел прочности на сжатие в 38 Мпа – 68 Мпа.