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EFFECT OF THERMAL TREATMENT ON PROPERTIES AND MICROSTRUCTURE OF POROUS ALUMINA

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ABSTRACT

This paper investigates influence of thermal treatment on the microstructure and properties of porous alumina-starch ceramics. The porous bodies were prepared through polymeric sponge method. The dried slurry of alumina and starch mixture was crushed and sieved on a 63 μm screen. The undersize particles were pre-sintered in the powder form at 1100, 1200, 1300, 1400 and 1500°C for 2 hours in an air furnace. The pressed forms of ceramic powder then sintered at 1500°C to produce porous alumina ceramic bodies with porosity from 25.9 to 31.2%. The modulus of rupture has successfully been improved about five times higher compared to the strength of porous ceramic body without presintering process. Besides that, the hardness value for double sintered porous ceramic was increased to 35.90 HV. SEM micrographs revealed better microstructural features of the ceramic body produced from pre-sintered samples. Based on the results obtained, pre-sintered process in the preparation of alumina-starch porous ceramics has managed to improve the hardness and strength of porous ceramic significantly. These ceramic bodies may have potential use in a wide range of applications due to an excellent combination of properties.

INTRODUCTION

Porous ceramics are expected to be used in a wide range of application, ranging from filters for gases and liquids, catalyst support, thermally or acoustically insulating bulk materials, coating layers or structural products [1]. Properties of a ceramic product are not only related to its mineralogical composition of raw materials but also to the different variables in the production operations: i.e. drying and shaping technique, sintering temperature, and thermal gradient, etc. [2]. Therefore, the preparation of porous ceramics needs thoroughly control of those parameters. Almost all ceramic materials have been made through the powder route, and also a variety of components used in modern technology has undergone sintering as one of the production steps. Sintering is one of the most important processes for the fabrication of ceramic materials [3]. The properties of ceramic materials can be modified through sintering process. The complexity of the sintering process makes this field a fascinating area for research.

The process by which small powder particles of a material are bonded together by solid-state diffusion is called sintering. In ceramic manufacturing, this thermal treatment results in the transformation of a porous compact into a dense, coherent product. In sintering process, particles are coalesced by solid-state diffusion at very high

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temperatures but below the melting point of the compound being sintered [4,5]. During the sintering of a powder compact, both densification and grain growth occur simultaneously [6]. In order to understand and control the sintering process, the relationship between densification and grain growth must be assessed. It concerns the materials constitutive laws governing the sintering process which is related to the microstructural evolution of the sintering component [7].

Usually, the preparation of ceramic body involves only single sintering process. The sintering process for ceramic preparation involves powder compaction method will be done after the pressing and drying process. However, in the present work, alumina ceramics which is prepared by powder route has gone through sintering process twice in order to produce porous alumina ceramic body.

METHODOLOGY

Ceramic bodies were made with alumina and starch powder with particle size below 63 μm. The mixture of raw materials based on volume percentage was milled for about 4 hours. Distilled water was used for preparation of slurries. The ground slurry was dried in an oven and sieved on a 63 μm screen. The undersize particles were pre-sintered at temperatures of 1100, 1200, 1300, 1400 and 1500 °C in the form of powder. The sintered ceramic powders were form into a green body by uniaxial pressing at 10 tonne to form rectangular bars. Polyvinyl alcohol was added during pressing in order to ease the sample fabrication process. The rectangular bars were then sintered in the air furnace at 1500 °C for 2 hours with a ramp of 2 °C/min. Figure 1 shows the controlled temperature profile of the furnace during heating and cooling of the raw mixture and the pressed bar samples. Meanwhile, the sequence of operation for preparing porous ceramic is depicted in Figure 2. Samples without presintering which undergone only single sintering temperature at 1500 °C were also prepared for comparison of data with presintering samples.

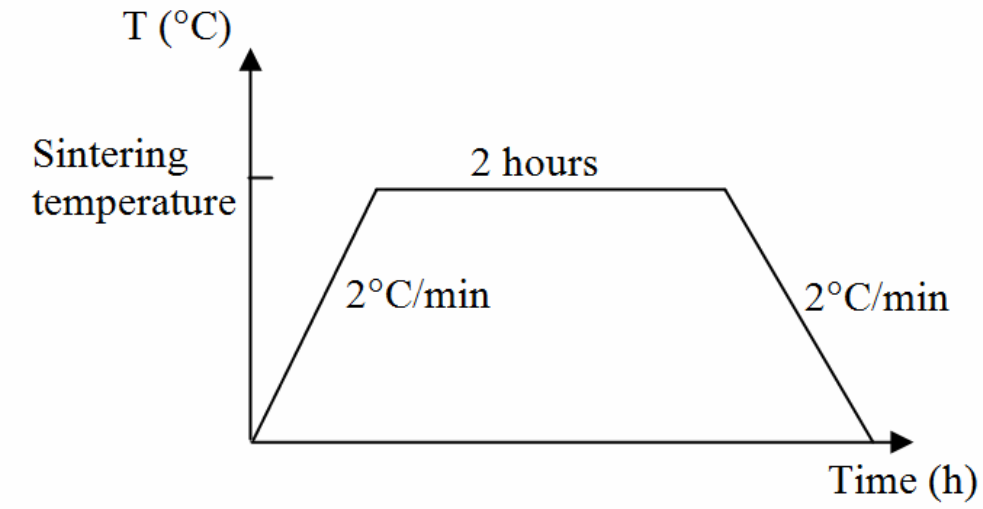

Figure 1: Sintering profile of porous alumina

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The sintered porous ceramic bodies were characterized by their physical and mechanical properties such as apparent porosity, density, flexural strength and hardness. Three-point bend strength was measured using Autograph AG-I Universal Testing Machine with applied load at a cross head speed of 1.0 mm/min. As for hardness test, the microhardness machine (HMV-2000 Shimadzu) was used. Apparent porosity and bulk density were measured by water immersion technique according to Archimedes' principal. Meanwhile, microstructural analysis of the sintered samples was conducted by using a Scanning Electron Microscopy, LEO model JSM-6380LA operated at 10-15kV.

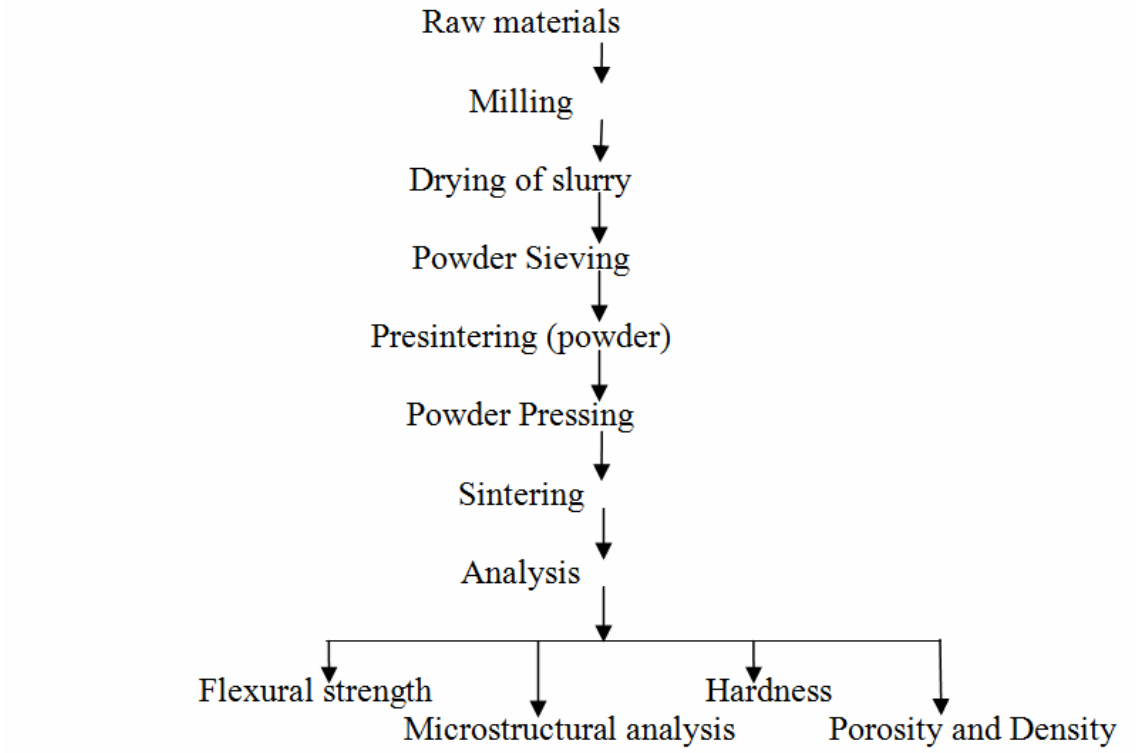

Figure 2: Flow chart for the sample preparation and testing of porous alumina

RESULTS AND DISCUSSION

Porosity and Density Analysis

Porosity is the dominant factor affecting the performance of ceramic materials. From the past research, the use of different starting alumina compounds, the choice of sintering temperature and hold time, as well as heating rate, and the introduction of sintering aid provide the opportunity to control the formation of pores size and porosity distribution in the wide range. The shrinkage during the sintering process tends to the simultaneous decrease of the porosity of the sintered samples [8].

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Table 1 show that porosity decreases with the increment of powder sintering temperature. The porosity drops gradually from 31.15% to 25.93%. This phenomenon is due to elimination of pores between aggregates and agglomerates and a fast grain growth during sintering. Cooled at lower temperature with longer dwelling time will make the porous ceramic body denser. Besides that, ceramic materials would like to get rid of all its grain boundaries so that it would have the lowest possible energy state.

Table 1: Porosity and density of porous alumina ceramics

	Powder sintering temperature °C (Samples from presintering process)					Samples without presintering
	1100	1200	1300	1400	1500	
Apparent Porosity (%)	31.15	29.01	28.03	27.55	25.93	39.29
Bulk Density (g/cm^3)	1.173	1.226	1.248	1.261	1.278	1.226

The result indicates that the cell size in ceramic foams is very sensitive to the baking condition and accelerates the cell coarsening [8]. Therefore, the controlling mechanism for porosity can be the alumina grain growth during firing.

In stark contrast to the characteristic of porosity, the bulk density of alumina foam increases with the rising of powder sintering temperature. The steadily rose of bulk density is caused by the declination of porosity in ceramic body. Samples without presintering have a bulk density of 1.226 g/cm^3. However, the same value of density is obtained from the ceramic powder which has gone through presintering at 1200°C. At higher presintering temperature of ceramic powder, the increment of bulk density can be achieved.

Flexural Strength

Figure 3 shows the graph of flexural strength versus alumina loading. Flexural strength for samples which undergo presintering process significantly exhibits higher value compared to samples without presintering. Alumina porous that has gone through presintering at 1500°C gave the highest strength 14.704 MPa which is almost five times higher than alumina porous without presintering with the strength of 3.096 MPa. The implementation of presintering process obviously contributes to drastic improvement of the strength of alumina porous body.

The changes in flexural strength can be related with the decreasing of porosity in sintered alumina body. Porosity has a significant role to influence the flexural strength of sintered alumina porous body. It is well known that the strength of the porous ceramics increases with the decreasing of porosity [9].

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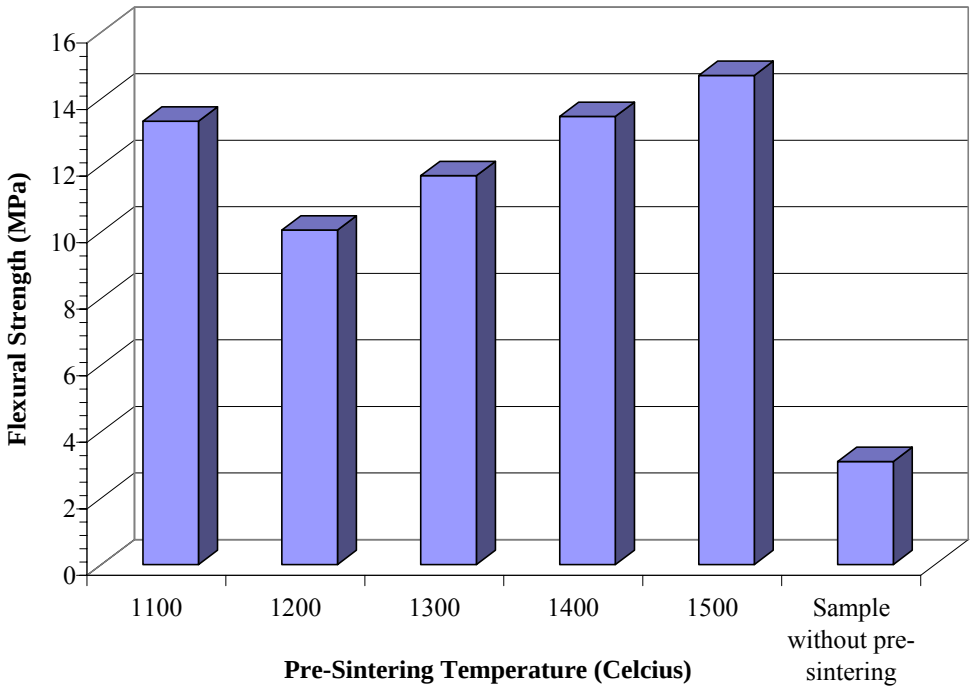

Figure 3: Flexural strength of alumina porous ceramic at different powder sintering temperature and sample without presintering

The effect of porosity on flexural strength for the sintered alumina foam is shown in Figure 4. The flexural strength is inversely proportional to the porosity [10,11]. Previous works indicated that changes of porous body strength as a result of grains grew up that led to the porous body had more contacting areas. It becomes the main reason for causing the change of porous alumina strength at that time.

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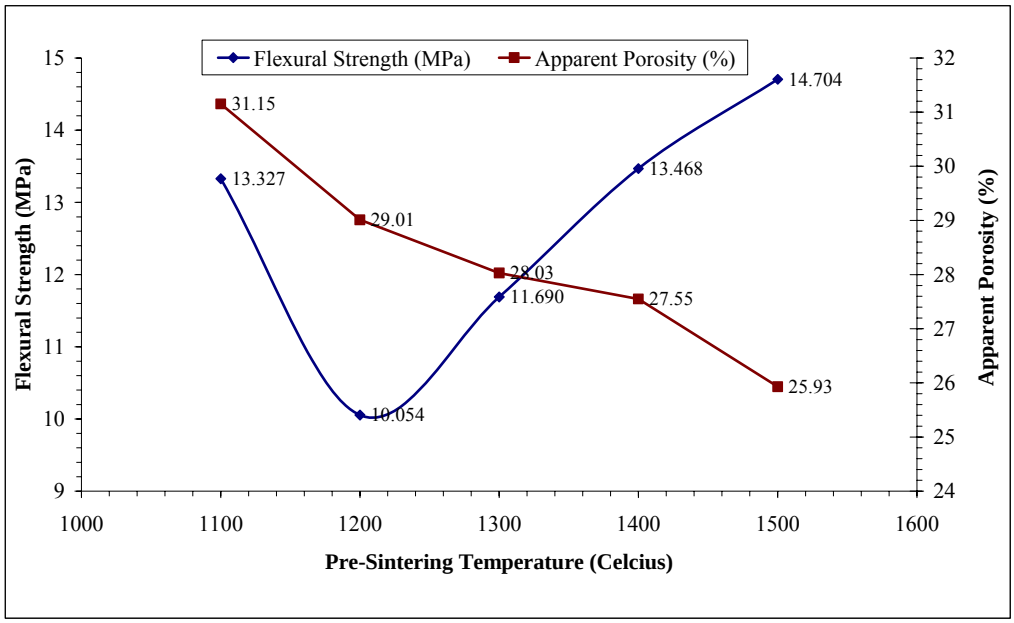

Figure 4: Correlation between flexural strength and porosity of sintered porous alumina.

Hardness

Results in Figure 5 depicts there is a wide gap between hardness value of sample with and without presintering process. Sample without presintering only gives 2.5 HV. Meanwhile, the hardness value for presintering alumina porous body has risen rapidly. This situation may be due to the powder refinement as the presintering temperature becomes higher. The decrement in porosity of porous body also contributes to the improvement of mechanical properties such as hardness.

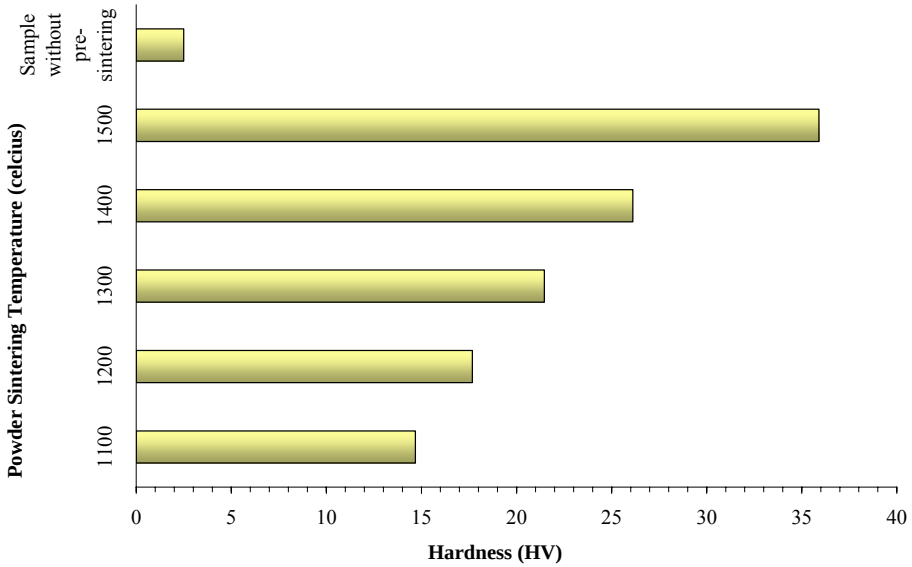

Figure 5: Hardness of alumina porous ceramic at different powder sintering temperature and sample without presintering

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Pre-sintered sample at 1100°C has successfully improved the hardness value to 14.68 HV which is about six times higher than the hardness of sample without presintering. Powder sintering at temperature 1100°C to 1500°C manages to rise up the hardness in the range of 14.68 HV to 35.90 HV.

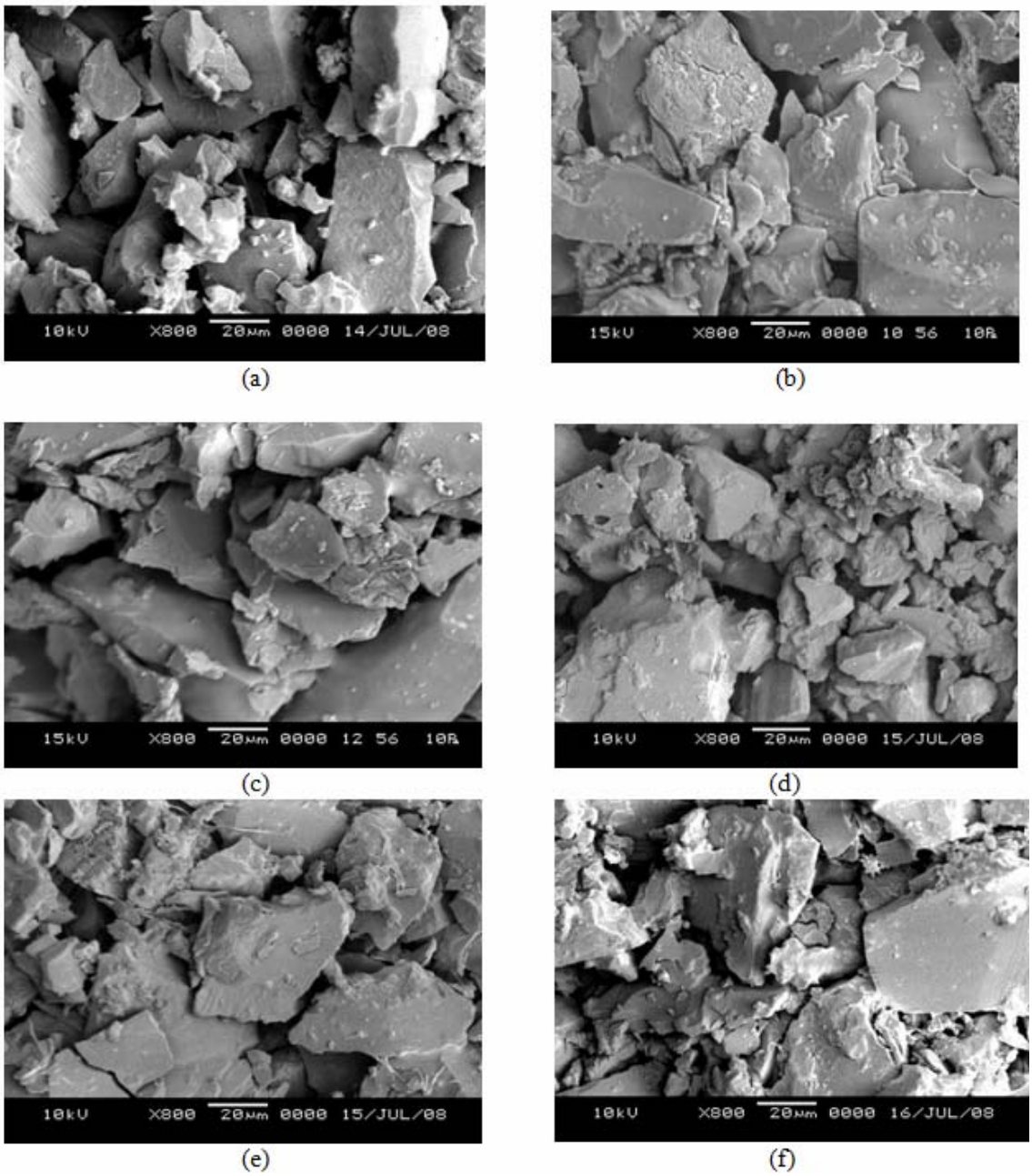

Figure 6: (a) Micrographs of single sintered porous alumina. Micrographs of porous alumina which has undergone presintering at (b) 1100°C (c) 1200°C (d) 1300°C (e) 1400°C (f)1500°C

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Microstructural Analysis

The SEM micrographs of the ceramic bodies (Figure 6) reveal better microstructural features of the ceramic body produced from the pre-sintered powder, having less pores and more particles with bigger size.

As the sintering temperature increases, the alumina interparticle necks are increased leading to more effective pore shrinkage [12,13]. Therefore, by rising the presintering temperature, has brought to a slight reduction in porosity which is the reason for causing the improvement in strength and hardness of porous alumina.

CONCLUSION

The influence of presintering on the mechanical properties and microstructure of porous alumina are investigated in this paper. Presintering of alumina powder with the addition of starch has proven to enhance the density, strength and hardness of alumina porous body significantly. Generally, the increment of presintering temperature has brought to the refinement of powder which contributes to the improvement of the porous alumina body properties. Presintering at only 1100°C enable the production of alumina porous body by press forming that gives 31.15% porosity with sufficiently higher hardness 14.68 HV and strength of 13.33 MPa. Such ceramic bodies may have a potential use in a wide range of applications due to an excellent combination of properties. The success can be explained by the presintering of mixtures in the powder form, as the sample without presintering produced much lower porous body characteristics.

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