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MANUFACTURING OF LARGE AND POLISHED CERAMIC PISTONS BY COLD ISOSTATIC PRESSING

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This paper discusses the challenges involved in producing high-quality alumina ceramics, particularly when it comes to achieving a suitable degree of surface roughness and high accuracy. Alumina ceramics are widely used in various applications due to their exceptional properties, including high strength, wear resistance and chemical stability. The alumina ceramic pistons with the desired properties and performance were produced from commercially available raw materials. To improve the densification and mechanical properties of the sintered ceramics, an organic binder was used. The use of a diamond abrasive slurry for polishing was also studied, which has proven to be particularly effective in achieving a high degree of surface finish. The study aims to produce alumina-based pistons with a low level of surface roughness as well as discusses the techniques and methods used to achieve this goal. The results of this paper include the porosity, mechanical properties and microstructure of the alumina ceramics produced in addition to the effectiveness of the techniques and methods applied.

Keywords: alumina ceramic pistons, advanced ceramics, reactive alumina

1. Introduction

Alumina ceramics are widely used in various applications due to their excellent properties such as high strength, wear resistance and chemical stability. However, production of alumina ceramic components with the desired properties and performance can be challenging, especially when it comes to achieving a high degree of surface smoothness and accuracy [1]-[2]. In the case of alumina-based advanced ceramics, especially where a particular spare part must fit precisely, the purity of the raw materials and the surface roughness of the finished product are particularly important. The surface finish of alumina ceramics can significantly affect their performance, especially in applications that require a low level of friction, tight tolerances and exceptional aesthetics [3]-[4]. Therefore, the development of effective polishing techniques for alumina ceramics has been extensively studied over the years. The aim of this study is to produce pressing granules that are suitably fine and develop an alumina ceramic-based piston with a low degree of surface roughness [5].

The production of alumina granules can be challenging as such materials are typically formed from fine powders that require careful processing to prevent agglomeration. During the production of pistons, it is particularly important to use appropriately homogeneous granules in order to determine the density, porosity and strength of the product [6]. One approach to overcome these challenges is the use of organic binders, which can help to form granules from fine powders. Organic binders are typically added to the alumina powders to improve the handling and processing characteristics of the materials such as strength, flowability and compressibility [7]-[8].

In addition to granulation techniques, isostatic pressing has been extensively studied to improve the properties and performance of ceramic materials [9], which is a technique that involves applying an equal degree of pressure from all directions on a ceramic powder or green body using a fluid medium to aid pressing and detachment of the mold. These agents optimize the production process by reducing friction between the granules as well as providing a separating effect between the pressing tool and the pressed material. Polyvinyl alcohol (PVA) is a water-soluble polymer that is used as a binder in isostatic pressing, which has proven to be effective in the production of ceramics [10]. Padmaja et al. studied the preparation and characterization of alumina ceramics using isostatic pressing with PVA as a binder. Their results showed that the addition of PVA improved the densification and mechanical properties of the sintered ceramics [11]. To separate the pre-press molds, polyoxyethylene-based organic additives are widely used, which thoroughly moisten the surface so the green body can be easily

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Table 1. Main properties of the base materials

Sample	Al_2O_3 content (wt.%)	Particle size (μm)	Specific surface area (m^2/g)	Na_2O content (wt.%)
A	99.8	0.3	7.5	0.1
B	99.6	2.0-6.0	0.7	0.1
C	99.8	2.0	0.7	0.1

removed from the surface of the pressing tool. Wang et al. used polyoxyethylene in order to produce appropriately shaped yttria-based advanced ceramics that exhibit a suitable degree of surface roughness [12]. The isostatic pressing process can significantly improve the density, homogeneity and mechanical properties of ceramic materials. The desired mechanical strength and porosity can be achieved by heat treating the green bodies. After sintering, the products must be polished to achieve a suitable degree of surface roughness and high accuracy [1-2, 10].

Alumina ceramics can be polished using various methods, including mechanical polishing, chemical polishing and electrolytic polishing. Among these techniques, mechanical polishing using abrasive slurries is the most commonly used method [13]. The use of diamond abrasives has proven to be particularly effective in achieving a high degree of surface finish. The diamond abrasive slurry polishing process involves the use of a rotating disk covered with a diamond abrasive slurry that grinds and removes material from the surface of the ceramic component. The surface finish achieved by diamond abrasive slurry polishing is significantly better than by other mechanical polishing methods [14]-[15]. Hudai et al. reported the polishing of sintered alumina ceramics using diamond abrasive slurries, who investigated the effects of various polishing parameters such as polishing pressure, polishing time and diamond particle size on the surface roughness and material removal rate from alumina ceramics. They concluded that the polishing pressure and diamond particle size both have a significant impact on the surface finish and material removal rate from alumina ceramics, moreover, that the use of diamond abrasive slurries can significantly improve and reduce the surface finish and surface roughness of alumina ceramics, respectively [16]. Hooper et al. investigated the polishing of high-purity zirconia ceramics using diamond abrasive slurries by comparing the surface finish achieved by diamond abrasive slurry polishing and mechanical polishing methods. They reported that the former significantly improved and reduced the surface finish and surface roughness, respectively, when compared to other polishing methods [17].

The purpose of this study was to produce alumina ceramic pistons with a purity of 99 % using commercially available alumina powders. The porosity, mechanical properties and microstructure of the products manufactured with the same production parameters were determined. According to previous results and the literature, the sintered products were polished with a diamond abrasive slurry in order to achieve a suitable degree of surface roughness.

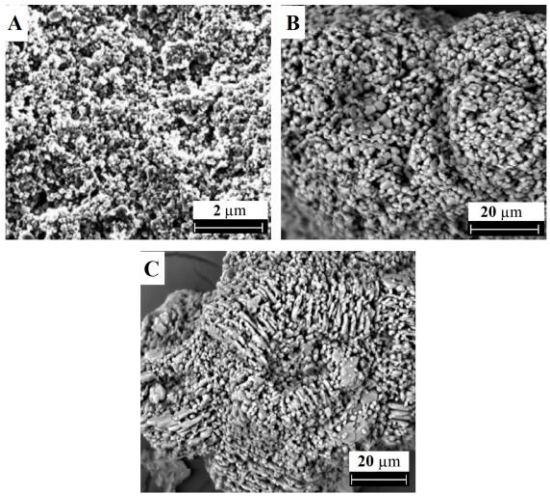

Figure 1. SEM micrographs of sample A (A – from the manufacturer's datasheet), sample B (B) and sample C (C)

2. Experimental

2.1. Fabrication of alumina ceramic pistons

Ceramic pistons were produced using high-grade calcined reactive alumina powders. Base material "A" was manufactured by Almatis GmbH., while type "B" was produced by Silkem Hungary Kft. and type "C" by Nabaltec AG. The main parameters of the raw materials are summarized in *Table 1*, which is based on the manufacturer's datasheet.

The microstructures of the alumina powders are illustrated in *Figure 1*.

The raw materials were ground in an industrial ball mill using ceramic balls with a diameter of 25 mm, where a grinding additive Dolapix PC21 (Zschimmer & Schwarz Chemie GmbH.) was added to the alumina powders. This grinding additive facilitates moistening of the granules and optimizes the grinding process. After 48 hours of grinding, a PVA-based organic binder (Optapix PA42, Zschimmer & Schwarz Chemie GmbH.) was added to the sludge at a rate of 2.0 wt.% of dry solids content per feedstock. The thermogravimetric curve in *Figure 2* shows that the Optapix PA42 organic binder started to decompose above 300 °C, which ensured that the sludge remained stable during spray drying and the green body did not crack during the heating phase of the sintering process.

The mixture was homogenized for 4 hours before the ready-to-press feedstock was spray dried. In an automated atomizer unit (Niro Atomizer D200), the slurry was fed at 21.4 bars and dried at 280 °C. The density of the sludge was 1.95 g/cm^3 during the drying process and the morphology of the granules after spray drying is shown in *Figure 3*.

Table 2. Density of the test specimens

Sample	Density after CIP (g/cm^3)	Density after sintering (g/cm^3)	Density after re-sintering (g/cm^3)
A	2.40±0.12	3.81±0.04	3.81±0.02
B	2.40±0.09	3.78±0.05	3.77±0.03
C	2.41±0.11	3.78±0.04	3.78±0.01

Table 3. Linear shrinkage values of the sintered samples

Sample	Transverse shrinkage (%)	Longitudinal shrinkage (%)
A	14.30±0.09	13.78±0.12
B	14.67±0.12	13.35±0.13
C	14.44±0.10	13.11±0.08

Table 4. Surface roughness values after various surface machining processes

Sample	Arithmetic mean surface roughness (*Ra*)	
	After grinding	After polishing
A	0.452±0.032	0.209±0.002
B	0.485±0.022	0.288±0.005
C	0.471±0.027	0.282±0.004

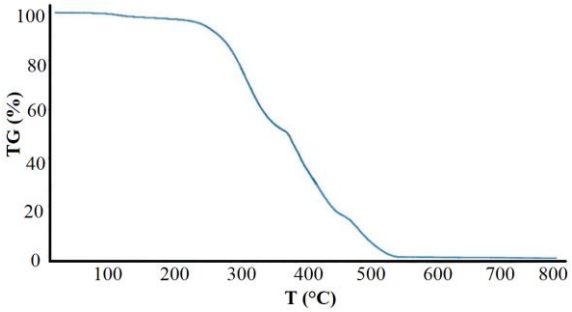

Figure 2. Thermogravimetric curve of the added Optapix PA42 organic binder

Figure 3. The shape of the particles formed after the spray-drying process

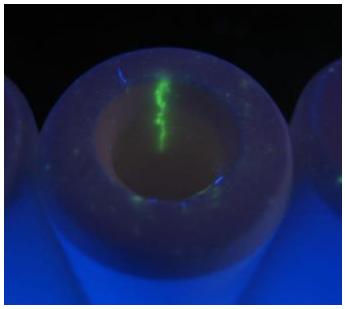

Figure 4. Defective product after penetration test

After the spray-drying process, a polyoxyethylene-based additive (ZUSOPLAST O 59, Zschimmer & Schwarz Chemie GmbH.) was added to the mixture at 0.5 wt.% to make it easier to separate the green bodies from the pressing tool. The coarse fraction of the granules was separated on a vibrating sieve. From the spray-dried granules obtained, the test specimens were pressed by cold isostatic pressing (CIP) using a densomatic isostatic press at a pressure of 1000 bars for 3 seconds. Regarding the isostatic pressing of large pistons manufactured for high pressure equipment, the decompression time is critical. Based on previous research results, the decompression time for all three types of alumina granules was 210 seconds.

After CIP, the test specimens were ground to the appropriate size and shape before being fired in a muffle furnace at 1550 °C for 4 hours in order to produce a completely solid product. To check the exact firing parameters, the products were re-sintered using the same parameters. If the density of the products is greatly reduced during the subsequent heat treatment, it can be concluded that the parameters set for the first firing were inappropriate. The density values of the pistons after isostatic pressing, sintering and re-sintering are summarized in *Table 2*. The results clearly show that after a single heat treatment the density of the pistons increased significantly and did not change during the re-sintering, i.e. the product was already sufficiently dense after the first sintering.

The linear shrinkage values after the first heat treatment of the test specimens are summarized in *Table 3*. Since the changes in the density and linear shrinkage of the products pressed from different raw materials were similar, no significant difference between the various granules was observed.

Following the sintering process, a penetration test was carried out using PFINDER 902 (Grimas Kft.), a fluorescent penetrant, to determine whether internal cracks were present. After being immersed, the samples were exposed to UVA radiation and the result obtained by examining the defective product is illustrated in *Figure 4*. Following the heat treatment, a negligible amount of cracking was observed on the products.

After sintering, the pistons were ground into their final shape and form before being polished by a Stahli FLM 500-R polishing machine for 16 minutes

using an abrasive slurry containing diamond particles of 3 µm in diameter. The average surface roughness (*Ra*) values resulting from grinding and polishing are summarized in *Table 4*. The results show that a finer surface roughness can be achieved on the pistons made of material type "A" by applying the same manufacturing and machining parameters. As reported in previous studies, the lower the *Ra* value, the better the tribological properties of the specimens [18]. This phenomenon is attributed to the fact that the average particle size of sample "A" (0.3 µm) was much smaller than for the other base materials (2.0-6.0 µm). According to the literature, polishing with a diamond abrasive slurry can produce a piston with excellent surface roughness properties on an industrial scale.

A picture of pistons of different sizes produced with the same technological parameters is shown in *Figure 5*. Products 42.0±0.1 mm in length and 18.0±0.02 mm in width were used for the measurements.

The products, produced with the same manufacturing and machining parameters as well as verified by penetration testing, were used for further tests.

2.2. Characterization

The microstructures of the raw materials and the granulates were tested by a FEI/Thermo Fisher Scientific Apreo LoVac scanning electron microscope and a Keyence VHX-2000 optical microscope.

Thermogravimetric measurements were taken by a MOM Q-1500 D thermogravimetric analyzer. The maximum temperature recorded was 800 °C, while the heating rate was 10 °C/min.

The densities of the pressed and sintered samples were determined by the well-known Archimedes' principle using water as the immersion fluid.

The surface roughness of the pistons was measured by a Taylor Hobson Surtronic S-128 profilometer with a total stroke length of 20 mm.

The compressive and bending strengths of the sintered samples were tested by an Instron 5967 dual column tensile testing machine at a feed rate of 144 kN/min.

The microhardness of the samples was characterized by a Vickers microhardness tester model Wolpert 402 MVD with a loading force of 1 kgf.

The internal structure of the samples was examined by X-ray computed tomography using a Nikon XT H 225. The evaluation was carried out using 2D images to determine their internal porosity and compactness.

3. Results and Analysis

3.1. Mechanical properties

The most important parameters of alumina ceramic pistons used in high-pressure equipment are their bending and compressive strengths. The results obtained by measuring the bending strength are summarized in

Figure 5. The sintered and polished alumina ceramic pistons

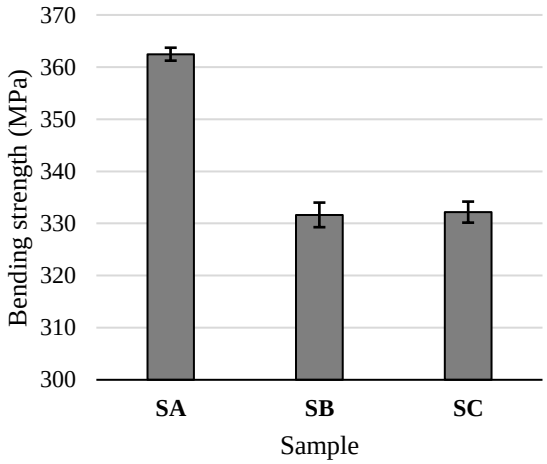

Figure 6. Bending strength of the sintered samples

Figure 6, where "SA" denotes the sintered product made from material type "A". The same marking was applied to the patterns on sintered products "SB" and "SC". The measurements were carried out at room temperature and in each case 5 parallel measurements were taken. The results show that alumina powder type "A" with a smaller particle size and larger specific surface area was able to produce a product with the highest bending strength and lowest standard deviation. This phenomenon can be explained by the fact that these particles are homogeneously distributed throughout the total volume of the product and fit more tightly than in the case of the other two raw alumina powders. Regarding alumina powders "B" and "C", a similar degree of flexural strength can be observed, which is associated with the same particle size distribution of the base material.

The measurements of the compressive strength were taken on the cylindrical surface of the piston as the instrument is capable of applying a maximum compressive force of 250 kN and the axial strength of the products is much higher than that. The compressive strength values are illustrated in *Figure 7*. In relation to the measurements made of the flexural strength, the highest compressive strength was achieved on the pistons produced using alumina powder type "A". The results of the strength tests show that the microhardness of pistons produced with the same technological parameters and machined under the same conditions is favorable when the average grain size and specific surface area of the starting material used is smaller and larger, respectively.

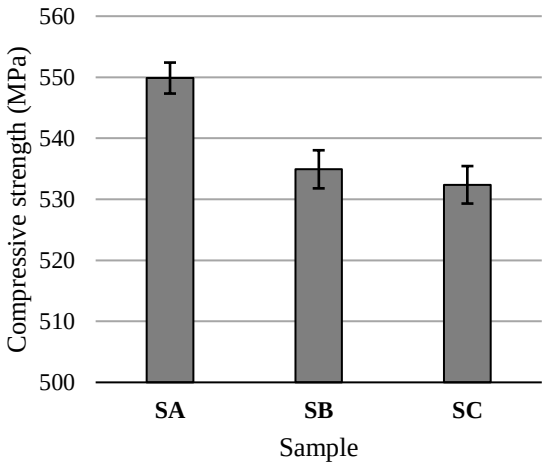

Figure 7. Compressive strength of the sintered pistons

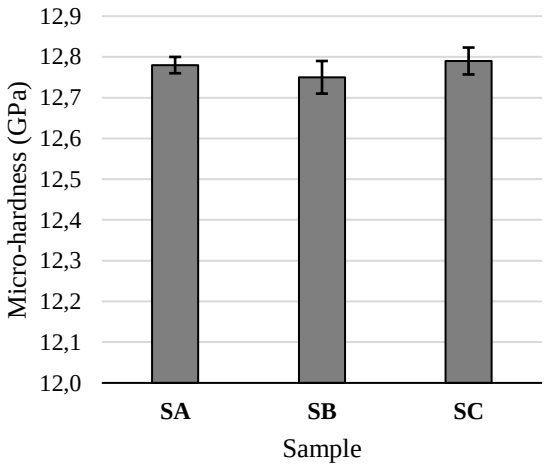

Figure 8. Microhardness of the pistons

The microhardness values measured on the surface of the pistons are illustrated in *Figure 8*. The microhardness of the samples made from different materials is almost identical, however, the standard deviations for samples “SB” and “SC” are higher. This phenomenon can be explained by the fact that the surface roughness of “SA” - which was more polished and the particle size of its starting material much smaller - is lower. Therefore, a denser product can be produced during the sintering processes.

3.2. Microstructure

The 2-dimensional X-ray computed tomographic images are illustrated in *Figure 9*. The tests were carried out after the sintering process. For the product manufactured from alumina powder type “A”, a compact structure is observed, that is, the sample does not contain large pores or cracks. However, in the case of the sample labelled “SB”, small pores and thin cracks are visible in the part denoted in blue. Such small cracks are not detectable by the aforementioned penetration test. In the case of the sample labelled "SC", no cracking was observed, however, a few small pores were visible in this sample which are highlighted in red. The decrease in strength observed in both samples "SB" and "SC" during the compression and bending tests is explained by the pores they contain.

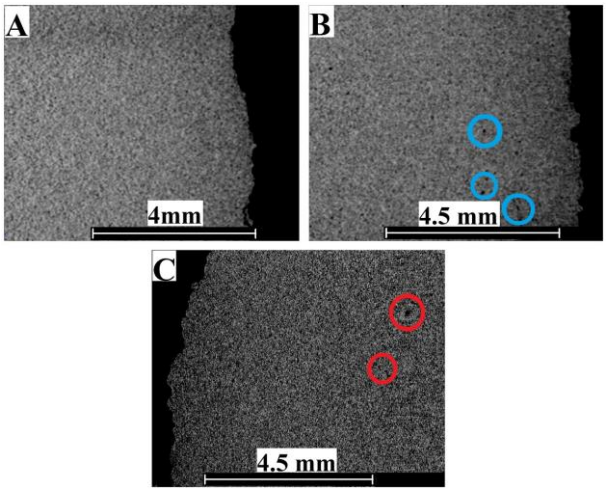

Figure 9. The internal microstructure of the sintered pistons: SA (A), SB (B) and SC (C)

4. Conclusions

This experimental work demonstrated that a sufficiently dense and polishable ceramic piston can be produced by adding PVA and polyoxyethylene as described in the literature. By applying the same manufacturing parameters, alumina from different manufacturers behaved in the same way, namely shrunk to the same extent during heat treatment and sintered to yield the same degree of compactness. However, during the final machining stage, a lower surface roughness was obtained by applying the same polishing parameters for the product produced from particles with a small average grain size and large specific surface area. The mechanical properties test showed that the strength of the product produced from raw material type “A” was best but the microhardness values were the same for all three raw materials. If a reactive alumina powder with a small average particle size and large specific surface area is used, a more polishable product with a low surface roughness is produced, therefore, a better fitting component can be manufactured. The structure of this piston is more compact and its mechanical properties better than those made from larger particles.

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