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Ceramic injection moulding process of alumina

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ABSTRACT

Purpose: The aim of this research is presented ceramic injection moulding process of alumina parts. Firstly alumina parts was performed by using binder system. The binder consisted of a mixture of a polypropylene (PP), paraffin wax (PW) and stearic acid (SA).

Design/methodology/approach: The volume fractions of powder in the feedstocks were changed from 40-50%vol and the volume of polypropylene were changed from 20-34%vol The concentrations of SA were kept at 6%vol. Secondly the feedstock was heated to melt the binder and injected into a mould. Thirdly the polymeric and wax binder was debinding by using solvent and thermal debinding. The thermal cycle was performed based on the results of the thermogravimetric analysis. Previously samples were sintered in one cycle with debinding of the binder during 23 h at 1400°C using heating rates of 0.5°C/min.

Findings: Thermogravimetric analysis (TGA) was performed to determine decomposition temperatures of polypropylene, paraffin and stearic acid. Morphology of alumina powder by scanning electron microscopy (SEM) was disclosed.

Originality/value: The paper presents ceramic injection moulding process of alumina parts for selected samples.

Keywords: Powder injection moulding; Aluminium oxide; Thermogravimetric analysis

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MANUFACTURING AND PROCESSING

1. Introduction

Powder injection moulding (PIM) is a new forming method for ceramics (CIM) and metals (MIM) materials. PIM process is a combination of the two technologies: plastic injection moulding and powder metallurgy. Powder injection moulding is possible thanks to the binding agent used, which is composed mostly from the thermoplastic polymers, and its portion is in the range 30-55%, depending on the powder used, shape of grains, wettability, specific surface, forming temperature, binding agent properties and other factors, which influence properties of

the powder slurry. Ceramic injection moulding (CIM) allows the manufacturing of complex three dimensional parts with narrow dimensional tolerances. Cost-intensive post-processing can often be avoided, which allows economical mass production of ceramic components. Repeatedly processing steps have to be controlled in order to produce valuable and fully functional products. Mould design and injection parameters highly influence properties of the finished product [1-8].

Unfavourable parameters of injection moulding or die will lead to products with defects such as voids, cracks etc. CIM uses ceramic powders such as alumina, titania, zirconia, etc. Unique properties of ceramic materials like a good mechanical properties, low specific weight and resistance to high temperature makes them interesting material for a wide variety of applications. These process is generally limited to parts less than 400 g, and suitable for the production of parts with complex geometry. Ceramic Injection moulding is used in several areas of industry like an automotive, medical and telecommunication industry [2-3].

The ceramic material is conventionally processed through a powder metallurgy (PM) process, consisting of [9,10]:

- powder production,
- mixing,
- moulding,
- debinding,
- furnace sintering.

A flowchart of the Ceramic Injection Moulding basic steps shown in Fig. 1.

In injection moulding of ceramic powders the powder is mixed with plasticizer. Plasticizer acts as a binder. The feedstock (powder and binder) is moulded using extruder machine similar to that used for polymer moulding. In extruder machine the feedstock is mixed to obtain a homogeneous mixture The composition of the powder-binder-mixture, the feedstock, is the most important factor due to the fact that it the processing conditions, i.e. mixing, flow and debinding behaviour [2,9].

A homogeneous mixture of ingredients known as feedstock is directly inserted into injection moulding machine and then injected into a mould. Then plasticizer is removed from component by thermal heating. The result is known as the porous component that still contains its original geometry and size. Injection moulding and sintering are the most important steps related to forming the final part [9,10].

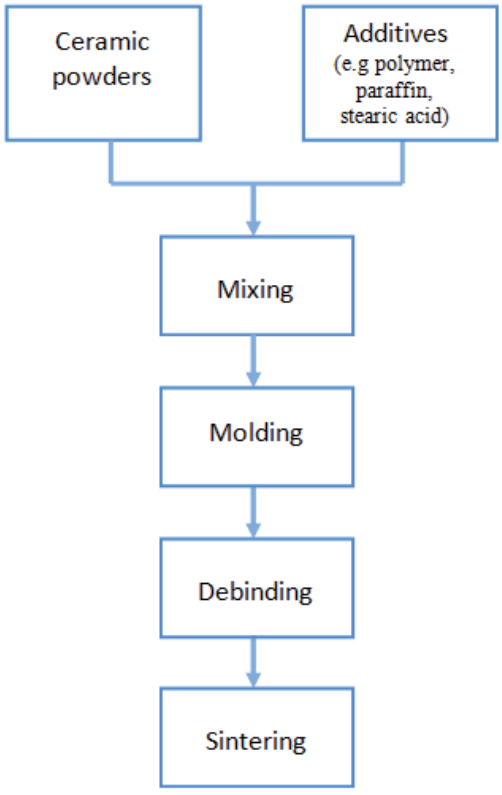

Fig. 1. A flowchart of the Ceramic Injection Moulding basic steps

2. Materials and methodology

2.1. Materials

The alumina powder produced by Nabaltec, NABALOX®, NO – 115.

Nabaltec produces calcined aluminium oxides for a wide variety of applications in the refractory and ceramics industry. The calcination process determines the a – Al_2O_3 content, structure, size and morphology of the primary crystallites. The required product properties are optimized and refined by grinding, air separation or granulation [11].

This provides NABALOX®-aluminium oxide with a number of advantages [11]:

- optimized packing density,
- wide sintering range,
- low sintering temperature,
- high sintered densities,
- flexible processing,
- optimized flow properties.

Nabaltec has a quality assurance process that is independent of the production process and as such is guaranteed and controlled, conforming to international standard classifications ISO 9001. Nabaltec is also certified according to ISO 14001, OHSAS 18001, ISO 17025 and has also a certified energy management [11].

NABALOX®, Aluminium Oxides, for the production of [11]:

- ceramic and refractory products,
- polishing and grinding media,
- catalysts,
- automotive components.

The alumina powder used in study has a medium *p* particle size of around 20 μm. The powder density was 3.9 g/cm³. To achieve favourable properties the binder consisted of a mixture of a polypropylene (PP), paraffin wax (PW) and stearic acid (SA). Stearic acid as a dispersant improve the dispersion of powder in binder and as plasticizer enhances the miscibility among binder components. Characteristics of binder components used in this study are shown Table 1.

2.2. Morphology

The morphology of the alumina powder was irregular in shape as shown in Figs. 2-3.

The alumina powder has a tendency to agglomerate because of the small particle size 2-19 μm as shown in Fig. 4.

2.3. Mixing

Four different feedstocks (mixtures of powder and binder) were prepared according to the compositions shown in Table 2. The volume fractions of powder in the feedstocks were changed from 40-50%vol and the volume of polypropylene was changed from 20-34%vol. The concentrations of SA were kept at 6%vol.

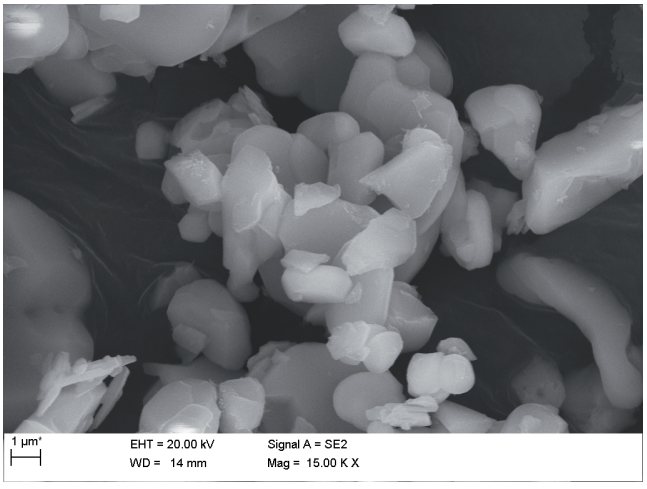

Fig. 2. Morphology of alumina powder by scanning electron microscopy (SEM); Magnification 15000x

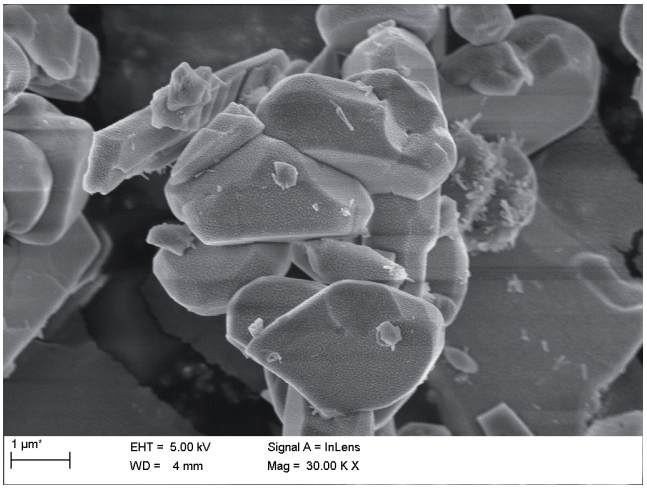

Fig. 3. Morphology of alumina powder by scanning electron microscopy (SEM); Magnification 30000x

Table 1.
Characteristics of binder components used in this study

Binder component	Density, g/cm³	Tm, °C	Decomposition temperature, °C
PP	0.9	130	280-450
SA	0.94	71.05	200-400
PW	0.91	56.98	250-342

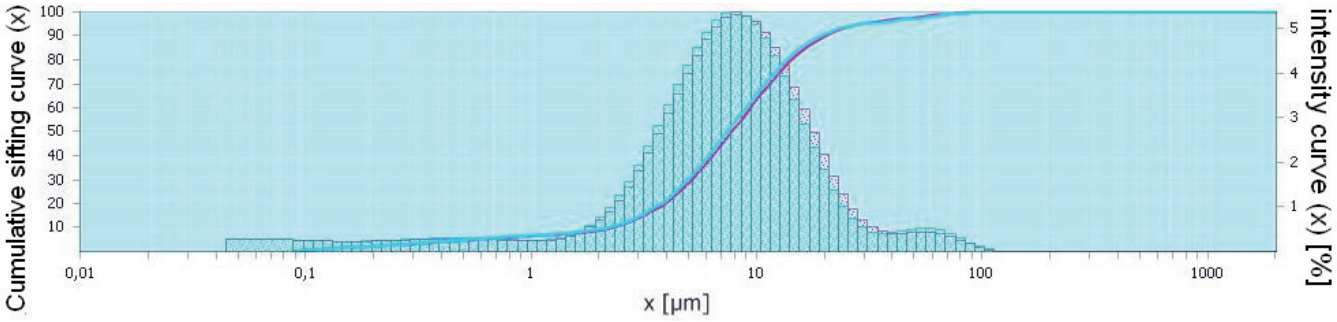

Fig. 4. The particle size distribution

Table 2.
Composition of feedstocks (vol%)

Feedstocks	Alumina		PP		PW		SA	
	%vol	%wt	%vol	%wt	%vol	%wt	%vol	%wt
F40/20/34/6	40	74.04	20	8.59	34	14.69	6	2.68
F40/27/27/6	40	74.06	27	11.59	27	11.66	6	2.69
F40/34/20/6	40	74.07	34	14.61	20	8.64	6	2.68
F50/22/22/6	50	81.06	22	8.27	22	8.32	6	2.35

Mixing process of alumina powder and binder was conducted in a Zamak Mercator extruder machine with a pair of rotating screws inside machine. The maximum capacity of the mixing chamber was 20 cm³. The torque value is a measure of the resistance on the rotating screws. The homogeneity during mixing can be predicted through torque curves.

Moulding step was optimized using torque measurements in a extruder machine at 155°C and 40 rpm. According to the thermal properties of the mixtures of powder and binder components the extrusion temperature (155°C) was higher than the highest melting point of the mixture but lower than the lowest degradation temperature of the binder mixture.

The feedstocks were extruder frequently to get homogenous mixtures.

Mixtures of powder and binder (PP, PW, SA) used in this study are shown Fig. 5.

Fig. 5. Mixtures of powder and binder (PP, PW, SA)

2.4. Injection moulding

Injection process was carried out in an Zamak Mercator injection machine. The injection moulding parameters were optimized by experimental method. Mould was cold and injection temperature was 150°C. Injection pressure was 2 bar.

The Fig. 6 shown an injection moulded part.

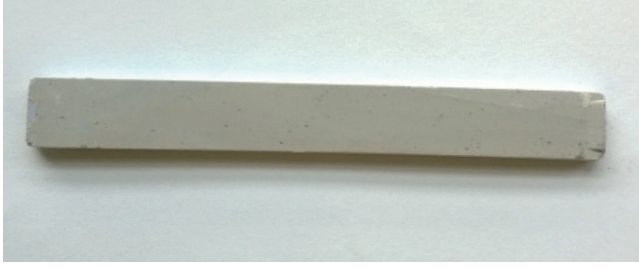

Fig. 6. Injection moulded part - F50/22/22/6

3. Results and discussion

3.1. Debinding and sintering

Debinding process was carried out after injection step. The organic part was removed through thermal and combination of solvent and thermal debinding. Samples were sintered at 1400°C in one cycle with debinding. The thermal cycle was performed based on the results of the thermogravimetric analysis. Thermal debiding was performed during 19 hours and 10 minutes using heating rates 0.5°C. Combination of solvent and thermal debinding was performed by dipping in heptane.

Solvent-thermal debinding was carried out in air. Total time of debiding cycle was performed during 15 hours and 18 minutes using heating rates from 0.5-4°C/min.

Debinding (thermal and solvent-thermal) schedule based on a feedstock F50/22/22/6 shown in Table 3.

Thermogravimetric analysis (TGA) was performed to determine decomposition temperatures of polypropylene (Fig. 7), paraffin (Fig. 8) and stearic acid (Fig. 9).

Table 3.
Debinding (thermal and solvent-thermal) schedule based on a feedstock F50/22/22/6

Thermal debiding	Heating rate, °C/min	Debinding temperature, °C	Debinding time, min
1	1	250	30
2	0.5	300	30
3	0.5	350	30
4	0.5	400	30
5	0.5	450	30
6	0.5	30	0
Solvent-thermal debiding	Heating rate, °C/min	Debinding temperature, °C	Debinding time, min
1	0.5	400	30
2	0.5	450	30
3	4	30	0

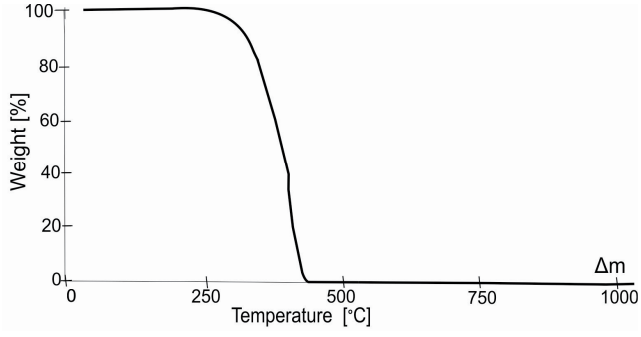

Fig. 7. Thermogravimetric analysis of polypropylene with heating rate 7.5°C to 1000°C in air

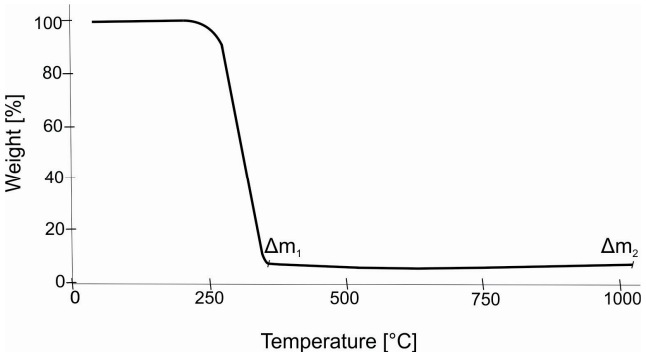

Fig. 8. Thermogravimetric analysis of paraffin with heating rate 7.5°C to 1000°C in air

Sintering was performed at 1400°C for 9 h, without the protective gas. After sintering, the theoretical density was examined by hydrostatic pressure. Was also calculated the total porosity and shrinkage.

Comparison of the theoretical density, apparent density, total porosity and shrinkage for F50/22/22/6 for thermal degradation and for solvent-thermal degradation shown in Table 4.

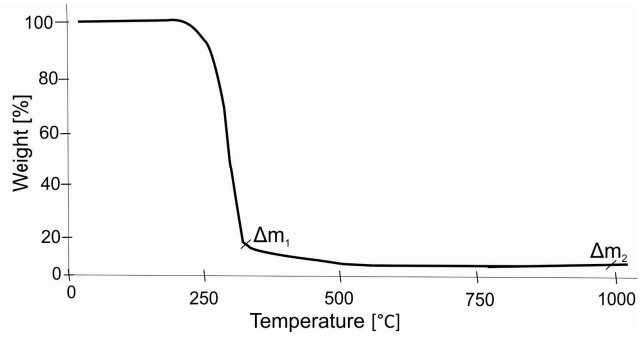

Fig. 9. Thermogravimetric analysis of stearic acid with heating rate 7.5°C to 1000°C in air

Table 4.
Comparison of the theoretical density, apparent density, total porosity and shrinkage for F50/22/22/6 for thermal degradation and for solvent-thermal degradation

	Sintering with thermal debinding	Sintering with solvent-thermal debinding
Theoretical density, g/cm³	3.9	3.9
Apparent density, g/cm³	1.97	1.74
Total porosity, %	55	49
Shrinkage, %	3	2

3.2. Conclusion

Mixing of PP and PW at 155°C was easy and prepared feedstocks the steady state were obtained quickly. Mixing of blend with PW content higher than 27%vol was difficult due to the low viscosity of this component at this temperature.

The addition of small quantities of SA to binder systems used in PIM is beneficial because substantially reduces the abrasion of the powder. Stearic acid as a surfactant reduces the contact angle by lowering the surface energy of the binder mixtures during injection moulding. allowing increase the solid loading and giving a better homogeneity.

The homogenization time for the mixture slightly increased with the powder loading as a consequence of higher resistance on the rotating screws.

Removing the binder was performed by using thermal and a combination of solvent-thermal debinding. The thermal elimination of binder was optimized by means of thermogravimetric analysis of the binder. which provides information on the degradation temperature range of the binder components. Debinding performed by dipping in heptane reduced sintering time of 3 hours and 52 minutes.

Binder components suitable for use in feedstocks for shaping alumina powders by ceramic injection moulding has been tested.

The possibility of degradation the both principal components of the binder in heptane permitted to use a solvent and thermal debinding process which reducing considerably the total debinding time and the formation of defects during debinding process.

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