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Low-Pressure Injection Molding of Alumina Ceramics Using a Carnauba Wax Binder: Preliminary Results

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Abstract. Carnauba wax, a natural product from Northeastern Brazil, has found application in the processing of ceramics. However, the use of pure carnauba wax is not recommended due to its narrow melting range and poor mechanical properties. In the present work carnauba wax based organic vehicles with the addition of low-density polyethylene and stearic acid were developed for use in the low-pressure injection molding of alumina ceramics. Viscosimetric testing was employed for the determination of optimal composition of the organic vehicle. The optimal content of ceramic powder in the mixture was also determined. All the materials used are easily available in the Brazilian market. A simple ceramic part was injected at low pressures (0.6MPa) using a semi-automatic injection molding machine. For this purpose a double cavity mold was designed and built. Preliminary results demonstrate the technical viability of the process using the organic vehicle developed.

INTRODUCTION

Production of ceramic thread-guides is an application of advanced ceramics in the large-scale manufacturing of small and complex parts. The most common processes employed for producing such parts are slip-casting, pressing, extrusion and injection molding [1]. Fabrication of ceramic parts by injection molding presents several advantages, particularly in the case of small-sizes and complex shapes: injection allows for automation and a larger variety of shapes; high dimensional accuracy and good surface finish are also important assets, since they can make it unnecessary for the parts to be machined, which is an expensive and damaging process [2]. Selecting a ceramic thread-guide among the various existing models is not a simple task: the choice depends on the thread characteristics (hardness and surface finish) and its operating parameters such as speed, tensile stress and temperature [1]. This work presents the different stages of the low-pressure injection molding process applied to the production of a relatively simple alumina thread-guide. Initially the general aspects of the process are described, with emphasis on the development of a suitable organic vehicle. Detailed information and processing parameters for the alumina part are then presented, with comments on certain peculiarities of the process.

CERAMIC INJECTION MOLDING

In this technique the ceramic powder is initially mixed with a fluid vehicle, which disperses the powder, to provide a homogeneous blend with rheological properties compatible with mold filling [3]. Subsequently the vehicle, usually an organic binder, should be removed without causing the disruption of the assembly of particles. The process comprises the following stages[2,4]: selection of materials, blending, injection, removal of the vehicle and sintering. The first four stages are the most critical ones, concerning the introduction of defects. They also involve features novel to the ceramics industry, and some basic knowledge had to be borrowed from polymer technology [4]. The first step in the process is to select the ceramic powder and the vehicle which has to be mixed with the powder to confer fluidity, thus allowing injection. Desired properties depend on a complex

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interaction of many powder and binder characteristics and a compromise between sometimes conflicting requirements has to be sought.

An ideal powder for injection molding should be easily injected with the help of a minimum amount of organic vehicle, achieving high green density and mechanical strength, and also allowing easy removal of the organic vehicle. Although the basic role of the organic vehicle is to confer fluidity to the ceramic mixture, it also affects the particle packing, agglomeration, mixing, rheology, molding, the removal stage, shrinkage, defects and final chemistry of the parts [4]. A vehicle for injection molding should also have the ability to change state in the mold cavity in order to resist stresses associated with ejection and handling [5]. Organic vehicle systems usually consist of a number of components which are carefully selected. Although the systems can be very complex, the components can be classified into one of four categories: major component, minor component, plasticiser and processing aids [6].

The major component determines the general range of final vehicle properties. It is responsible for conferring fluidity to the powder, wetting the ceramic to remove air, accommodating change of state and giving strength to the green body. It should also leave low burn-out residue and be as cheap as possible. The minor component is often a thermoplastic or oil which is removed easily during the removal cycle. It is believed that this generates pore channels which allow easier removal of the other component. The plasticiser should have low volatility and mix well with other components to lower the viscosity of the ceramic-organic vehicle mixture. Finally, processing aids could also be added to the system to act as surfactants to the ceramic powder to improve the wetting characteristics and also to enhance deagglomeration, reduce melt viscosity and allow easy mold release.

The selection of a suitable organic vehicle for a given ceramic powder is still done empirically. The two main criteria for this selection are: it should provide a rheology that allows defect-free molding, and should be amenable to successful (or defect-free) removal. In order to meet the first criterion, extensive research in rheology has been done. However, the available guidelines are mostly qualitative and semi-quantitative [4]. The desirable features for the organic vehicle depend on several parameters, such as molding temperature, shear rate, solids content, ceramic powder characteristics and the presence of active surfacting agents. For a given solid content, a low-viscosity mix depends basically on the use of a low-viscosity vehicle, which is typically a wax [5]. Besides conferring a low-viscosity, the organic vehicle should also inhibit both separation and agglomeration of the powder. Since waxes are, in general, inadequate for such tasks, they are usually mixed with polymers, which also add to the green strength of the molded body. Rheology studies are therefore, needed in order to evaluate the adequacy of a vehicle for use with a given powder [4].

The injection stage is a direct legacy from polymer technology. Although the principles are similar to those employed in the injection of plastics, the practice of injection molding ceramics presents its own challenges: tooling has to be designed to cope with abrasive materials and brittle pieces, and the processing parameter must be carefully chosen to suit the particular blend to be injected. Some of these problems can be overcome, however, in the low-pressure processes, since the machines are smaller and of easier operation. Machine and tool wear are also minimized.

The removal stage involves extraction of a substantial amount of material, in the range of 30-40% vol., from the molded body. This is the most critical stage regarding the introduction of defects, and is also one of the least understood aspects of the injection molding of ceramics [2,4]. Removal of the vehicle can be achieved either by chemical or thermal methods. Pyrolysis is undoubtedly the most used method for removal of organic vehicle, both for ceramic and metal injection moldings

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[4,7]. Capillary action requires the use of a wicking material that is put in contact with the molding and provides for capillary flow at temperatures above the organic vehicle softening point [8]. In fact, it is common practice to lay specimens on an inert powder bed during removal of organic vehicle in both thermal and oxidative degradation methods [4,8].

EXPERIMENTAL DETAILS

In the production of ceramic thread-guides, the materials commonly employed are alumina (Al_2O_3) and titania (TiO_2) [1]. In the present work we used a calcined alumina (APC 2011SG, ex Alcoa do Brasil), the main characteristics of which are shown in Table 1. Particle shape was investigated by scanning electron microscopy Phillips XL-30. Samples of powder were dispersed in acetone and then allowed to dry on microscope stubs. They were carbon coated to avoid charging. Fig. 1 shows a picture of the powder. The particles exhibit a flake shape and are agglomerated.

Table 1. Chemical composition of the ceramic powder according to ALCOA do Brasil.

Component	**%**
Al_2O_3	99.4
SiO_2	0.04
Fe_2O_3	0.04
Na_2O (total)	0.11
Humidity (300°C)	0.2
Fire Loss (300 – 1100°C)	0.1
Mean Particle Size	2.6μm
Specific Surface Area	$1.4 mm^2/g$

Figure 1. Photography of the powder (3500x).

Carnauba wax type 1 (from Açu – RN, Brazil) was the major component of the vehicle used. The main characteristics of this material have been discussed elsewhere [9]. Low-density polyethylene (HI 865, ex OPP Poliolefinas, S.A.) was added as the minor component, to increase the green strength and widen the melting range of the vehicle. Stearic acid (ex Vetec, Quimica Fina, LTDA) was the surfactant employed. Relative proportions of the components were determined by viscosity measurements. The same procedure was adopted for determination of the powder content in the mixture.

Viscosity measurements were performed in a Brookfield LV-DVIII viscometer. A small quantities mixer was developed for the organic vehicle studies. The components were weighed and mixed at 145°C during 30 minutes. Several samples containing different amounts of the components were tested in the viscometer under various shear rate and temperatures. Those compositions which achieved the lowest viscosities were used to prepare a ceramic mix with different amounts of

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ceramic powder. Mixture was achieved in the mixing tank of the injection machine at 140°C. during 30 minutes under vacuum. Small samples of the mixes were taken from the tank and tested in the viscometer at 140°C for different shear rates. After viscosity measurements the various combinations of organic vehicle and powder were submitted to trial injection moldings. Depending on the ability to fill the mold without creating macrodefects, the mix and/or the vehicle could be reformulated, and the whole procedure repeated.

A small thread-guide with an "A" shape was chosen as the part to be molded. Fig. 2 shows mold details as well as the injected pieces. Tool material is SAE1020 plain carbon-steel.

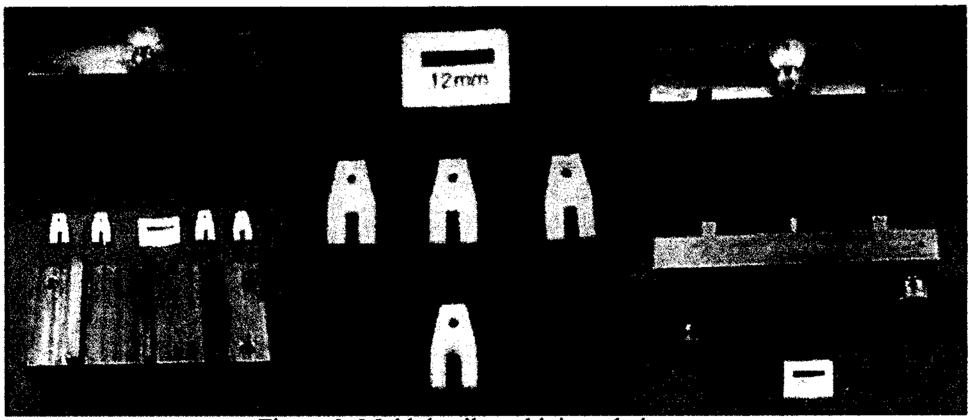

Figure 2. Mold details and injected pieces.

The machine used for injection in this work is a semi-automatic Peltsman MIGL33. Molding procedure is fairly simple and straightforward. The main parameters for the process: molding temperature of 140°C (to avoid degradation of vehicle components), injection time of 15s and a pressure of 0.6MPa.

Oxidative degradation was the method employed to remove the vehicle from molded bodies, although wicking also helped in the process. Each sample was put in a crucible and was completely covered by alumina powder. The crucible was put in a controlled muffle furnace in air and was heated according to this schedule: room temperature to 70°C at 0.5°C/min; it was kept for 24 hours at that temperature; 70°C to 380°C at 5°C/h; 24 hours at that temperature; 380°C to 750°C at 5°C/min; let the furnace cool down. This schedule was based on previous thermogravimetric studies [9,10] which evaluated the behavior of the vehicle upon heating. Since this is the most critical stage of the whole process, an iterative approach was adopted: parts which had been successfully molded from different mix formulation were inspected after being subjected to removal. This gave a feedback to the formulation stage, for some defects can only be detected after vehicle removal [4].

After a successful removal of the organic vehicle, the sample was carefully taken to a high-temperature sintering furnace, where it was heated at 300°C/h up to 1,650°C and then left at that temperature for one hour.

RESULTS

Fig. 3 shows the results of viscosity measurements at 140°C for three different compositions of organic vehicle suitable for low-pressure molding of powder parts. Several other formulations were tested, but they were not successful at posterior trial moldings. A higher amount of wax made the mixture easier to be injected, but more polyethylene helped in preventing collapse of samples during vehicle removal. Therefore, a compromise was made, and a composition of 94%wt carnauba, 5%wt LDPE and 1%wt stearic acid was adopted.

Fig. 4 presents results of viscosity measurements of the three different compositions mixed with the alumina powder. In the case of the mixture, the more ceramic powder the better, since this reduces the time spent in removing the vehicle. Besides, higher solids content reduces contraction and increase green-strength. On the other hand, more ceramic powder means higher viscosity and more difficulty with mold filling. Once again, a compromise is to be sought. For the research work described here, a composition of 80%wt alumina and 20%wt organic vehicle was apparently successful, since no macrodefects were noticed. Sintering was also performed without damaging the samples. Density of sintered parts was measured using the Archimedes principle. An average value of 3.7g/cm^3 was found, which is compatible with alumina samples sintered in air without using any sintering aids [2].

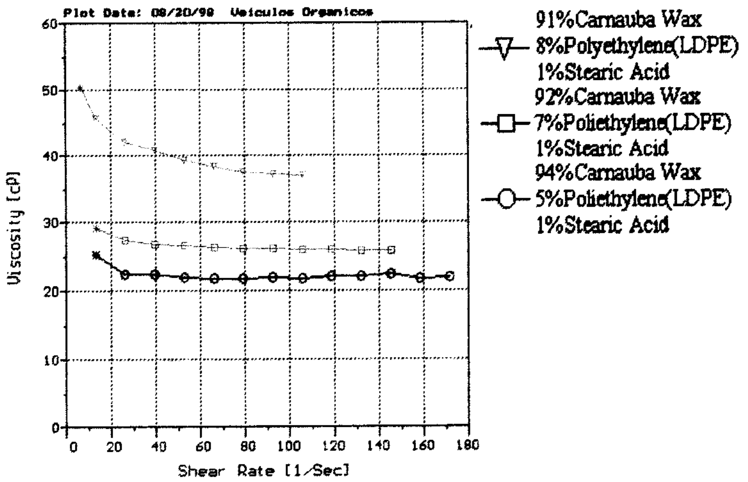

Figure 3. Organic vehicle viscosities at 140°C.

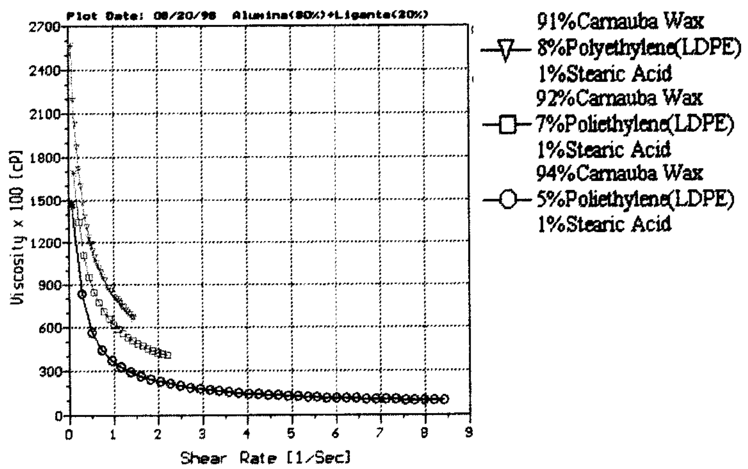

Figure 4. Mixture viscosities at 140°C.

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CONCLUSIONS

Technical viability of low-pressure injection molding of ceramics using materials produced in Brazil was demonstrated. The organic vehicle developed employing carnauba wax confirms the potentiality of this wax as a major component for organic vehicles. Low-pressure injection molding allows for the use of low-cost and relatively simple molds. Finally, the preliminary results show that injection molded parts are comparable to those obtained by other processes, with the additional advantages of flexibility and automation.

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