Congresso Brasileiro de Engenharia de Fabricação
02 a 04 de abril de 2001 • Curitiba - Paraná - Brasil

PRODUCTION OF COMPLEX PARTS BY LOW-PRESSURE INJECTION MOLDING OF GRANITE POWDERS.
Part I – Preparation of feedstock, injection and debinding

Paulo César G. Felix
Federal University of Ceará (UFC), Department of Mechanical and Production Engineering
pcfelix@dem.ufc.br – Fortaleza, CE, Brasil
Philip Frank Blazdel
Federal University of Ceará (UFC), Department of Mechanical and Production Engineering
philip@dem.ufc.br – Fortaleza, CE, Brasil
Ricardo Emílio F. Q. Nogueira
Federal University of Ceará (UFC), Department of Mechanical and Production Engineering
emilio@dem.ufc.br - Fortaleza, CE, Brasil

Abstract. *During the quarrying of the ornamental stones in Ceará state (Northeastern Brazil), a great amount of waste material is produced. Since most of this powder has not found economic use to date, it is simply, discarded becoming a potential environmental hazard. This paper describes the use of granite powder as feedstock for powder injection moulding, thus replacing costly ceramic materials. Initially the selected powder was milled and sieved. The resultant fines were then characterised by x-ray diffraction and scanning electron microscopy and the particle characteristics were also determined. The characterised material was mixed with a low-viscosity organic vehicle comprised of carnauba wax, low-density polyethylene and stearic acid. Rheology studies were performed in order to determine the optimum powder content compatible with the low-pressure injection moulding process, and the mixture was injected. Small thread-guides were produced using a two-cavities mould manufactured from low-carbon steel. Thermal analysis was employed to determine the appropriate debinding schedule. Investigation of both as-moulded and debound parts showed that the granite powder has a great potential as a substitute for more expensive ceramic materials.*

Keywords: *Injection, Molding, Granite, Ceramics.*

1. INTRODUCTION

Ceara State, in North-eastern Brazil, has great reserves of granite, which are commercially exploited and find use mainly as ornamental stones or in pavements(Vidal et al, 1999). Approximately 60% of the material is lost during processing, that is, from the quarry to the final cutting and polishing operations (Castelo Branco, 1998). The vast majority of those losses occur during the extraction or quarrying process, resulting in the accumulation of a substantial amount of fragments and powders. Besides the evident waste of raw material, the rejects are prone to cause serious damage to the environment, since they are simply discarded on land or even in riverbeds close to the mines. Therefore, reducing the environmental damage is very important for the mining companies. Depending on the kind of material and on the mine situation, there are a few succesful cases documented of re-use of rejects for the production of brita, paving stones and curb stones; and even tiles, using small stone-cutting machines (Braz e Amaral 1996). Brasileiro (1999) reports on the use of a residue from the cutting and polishing operations, the so-called sawing abrasive pulp or mud, that is transformed into blocks and construction bricks. The blocks can be used to build schools, hospitals and low-cost houses.

Granite is a very hard material, due to the minerals that compose it: quartz and feldspar. Due to its great hardness granite is difficult to work and so is an expensive building material. In this paper, the word granite is used in the commercial sense, which includes any non-calcareous rock amenable to polishing and which can be used for covering walls and floors (NITES Report, 1991). Such definition encompasses true granite rocks, plus other rocks like diorites, gabbros and meta-conglomerates . The objective of the research described below is to exploit the residual granite powder as a raw-material for the production of ceramic parts by the process of low-pressure injection moulding

Amongst ceramic fabrication routes, injection moulding stands out as one of the few processes capable of producing complex shapes in a automated fashion without the need for machining of sintered parts. In this technique the ceramic powder is mixed with a suitable vehicle or binder which disperses the powder and thus provide a homogeneous blend with rheological properties compatible with mould filling. Binder systems usually consist of a number of organic components which are carefully selected to confer optimum properties. After moulding the vehicle is removed and the part is sintered (Nogueira, 1992). Whereas conventional injection moulding employs very high injection pressures (from 70 to over 100MPa), in the low-pressure process the material is injected using pressures ranging from 0.35 to 0.7MPa. The main advantages of low-pressure injection moulding include reduced wear of the machines and moulds, less stress in the parts and less deflections of the moulds. All these factors allow the use of smaller and cheaper machines and moulds. However, the mixture to be injected must have a low viscosity, which restricts the choice of organics and limits the powder loading in the mixture (Mangels, 1994).

The powder to be injected must be tailored in order to allow a high solids content to be incorporated into a continuous binder matrix. The main characteristics of the suitable powder for injection molding have been listed elsewhere (German and Bose, 1997). The binder is a temporary vehicle used to confer fluidity to the mixture, pack the powder into a homogenous shape and thus keep the arrangement of particles until the beginning of sintering (German, 1989). It also affects several aspects of the process, such as injection, the removal stage, shrinkage, formation of defects and the final chemistry of the parts (Nogueira, 1992)

Selection of a suitable binder for a given ceramic powder is still done empirically. It should provide a rheology that allows defect-free moulding, and should be amenable to successful (or defect-free) removal. The desirable features for the binder depend on several parameters, such as moulding temperature, shear rate, solids content, ceramic powder characteristics and the presence of active surfactant agents. For a given solid content, a low-viscosity mix depends basically on the use of a low viscosity organic vehicle, which is typically a wax (German and Bose, 1997). Besides conferring a low-viscosity, the binder 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 moulded body. Rheology studies are therefore needed in order to evaluate the adequacy of a vehicle for use with a given powder (Nogueira, 1992)

After mixing and moulding, which in the case of the low-pressure process can be done in the same machine, the next step is binder removal. This is by far the most critical stage in the injection moulding of ceramics (Hwang, 1996). It involves extracting a large amount of material (in the range of 30 to 40% volume) from the moulded body, either by chemical or thermal methods. Extreme care has to be taken in order to avoid the introduction of defects (Nogueira,1992; Nogueira et al, 1998), what calls for long times and increases production costs. Moreover, many defects originated at previous stages only become evident after binder removal, such as non-homogeneous mixing, use of inadequate moulding parameters and defects in mould design.

Pyrolysis of the organic vehicle, either by evaporation, thermal degradation or oxidation is undoubtedly the most used method for the removal of binder from ceramic mouldings. It is common practice to use commercial furnaces both in air and controlled atmospheres (Mutsuddy, 1991). It is also common to lay specimens on an inert powder bed during debinding, which prevents distortion and sagging, and also accelerates the removal by capillary action (Hwang, 1996). Removal of binder by pyrolysis, in particular in the case of thick-walled parts, depends strongly on controlling the rate of decomposition of the binder system (Edirisinghe, 1991). The aim is to allow for the outward diffusion and evaporation of decomposition products to occur without the formation of any gas which would cause the formation of voids. Considering the potential for forming defects from both thermal and chemical removal of organic vehicles, another option is the use of supercritical debinding. This technique is currently being developed at the UFC for the low-pressure injection moulding of ceramics and will be presented in a following paper. Following debinding sintering is performed.

2. MATERIALS AND METHODS

In order to assess the potential of granite powders as a replacement for conventional ceramics, it was decided to use it for the moulding of a ceramic thread-guide. In the production of ceramic thread-guides used in the textile industry, the materials commonly employed are alumina and titania (Macéa et al, 1989). The granite powder selected was Granito Asa Branca.

The material was ball-milled for 24 hours and classified by sieving with a minimum screen aperture of 80μm. It was characterised by X-ray diffraction and scanning electron microscopy. Its chemical composition was then compared to the petrographical analysis of the original material. Specific surface area was determined using the BET method.

The organic vehicle used emplyed carnauba wax as the main component (94% wt) plus small additions of LDPE (5% wt) and stearic acid (1% wt). More details on this vehicle are given elsewhere (Nogueira et al, 1998; Lima Filho *et al*, 2000). Rheometric studies determined the optimum composition of the mixture powder-organic vehicle. Binder viscosity was determined with a Brookfield LV – DVIII rheometer, varying the applied shear rates and temperatures.

Mixing and homogeneization of the ceramic blend was performed with the aid of a double-blade planetary mixer, available in the moulding machine (semi-automatic model MIGL 33 ex Peltsman Corp.), during 30 minutes under vacuum at 140^{o}C, to eliminate humidity and avoid formation of bubbles. The mixture was then injected in a two-cavitie mould under a pressure of 0.6 MPa. Injection procedures are very straightforward and have been described elsewhere(Nogueira et al, 1998).

Binder removal was undertaken in a muffle furnace under normal atmosphere. The parts were laid on a alumina powder bed. Heating schedule was determined by thermal analysis. The parts were sintered in air at 1100^{o}C for 90min with a heating rate of 1^{o}C/min. Comparison of dimensions before and after sintering was performed to determine shrinkage.

3. RESULTS

Figure 1 shows the result of the X-ray analysis. Phases present are: Albite - $NaAlSi_3O_8$, Quartz - SiO_2 and Microcline – $KAlSi_3O_8$. Comparison with the petrographical analysis of the original rock is shown in Table 1.

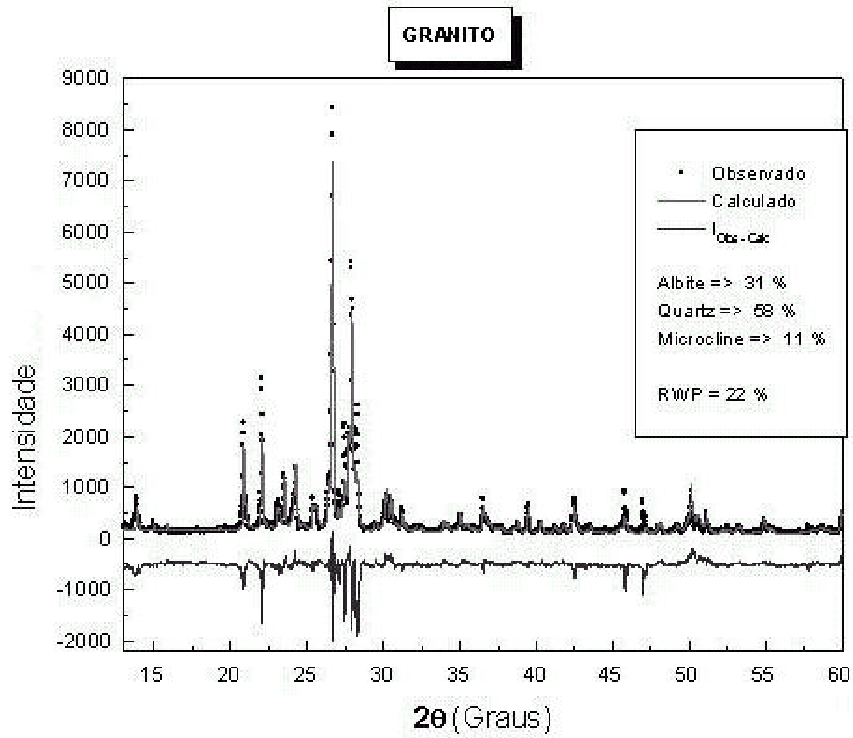

Figure 1- Diffractogram of "Asa Branca" Granite

Table 1: Composition of Asa Branca Granite

	Albite	Quartz	Microcline	Non-essential minerals
X-ray	31 %	58 %	11 %	–
Petrographical Analysis	33 %	49 %	10 %	8 %

The small difference in the results is probably due to the particularities of each method and is well within the resolution of the techniques.

Scanning electron microscopy was done to examine particle morphology. Fig. 2 is a micrography of the milled and sieved powder. The particles are not angular, but smooth and the particle size distribution is wide, ranging from 10 to 120µm. Further improvements are possible with increased milling times.

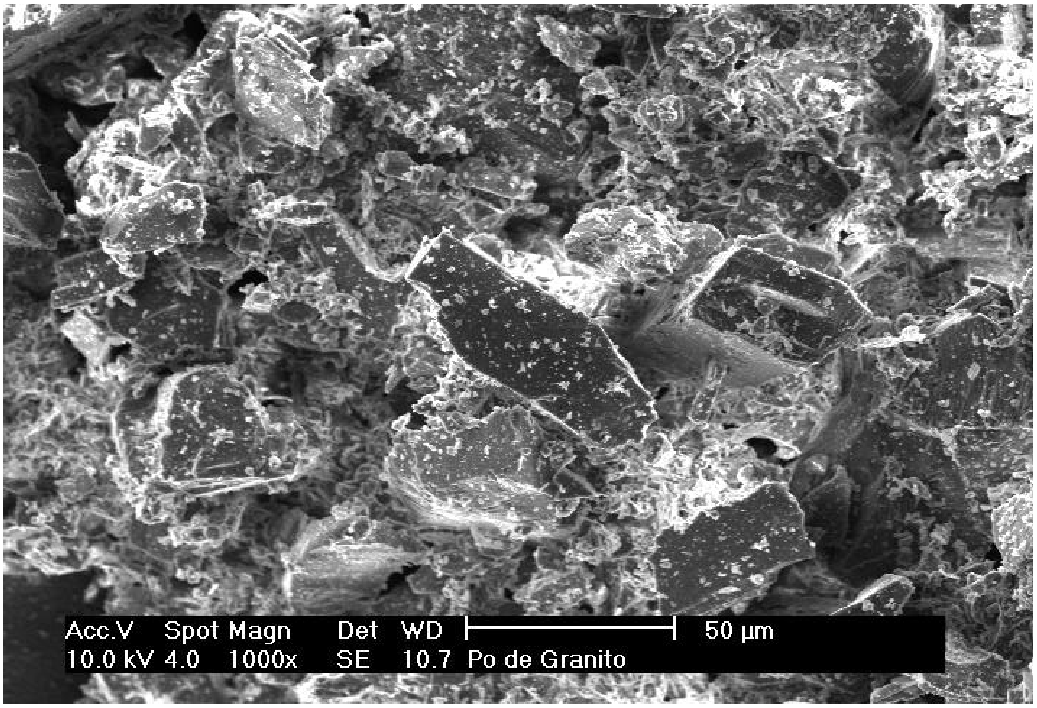

Figure 2 - Morphology of granite powder

Viscosity measurements showed that the optimum granite powder content in the mixture to be injected was 82% in weight (approx. 62% in volume) at the injection temperature of 140^oC. This provided a high solids loading with a low-viscosity, as seen in Figure 3.

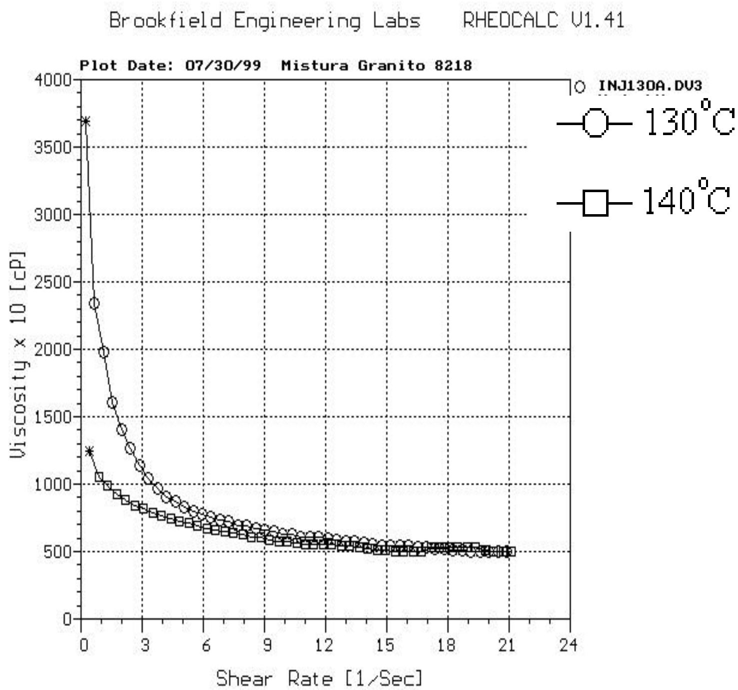

Figure 3**.** Mixture 82% powder-18% binder

Figure 4 shows the injected granite thread-guides, before binder removal (left), after binder removal (center) and after sintering (right). Binder removal was achieved without introduction of macrodefects, according to a very careful schedule with 23 steps and which took 96 hours to be completed. This is obviously unacceptable for economical reasons, and work is being done in order to develop an organic vehicle that can be removed by supercritical debinding. Sintered parts contracted approximately 8.0% when compared to the injected ones, and exhibited a glazed appearance with smooth (round) edges. Mechanical properties and final densities are still being measured.

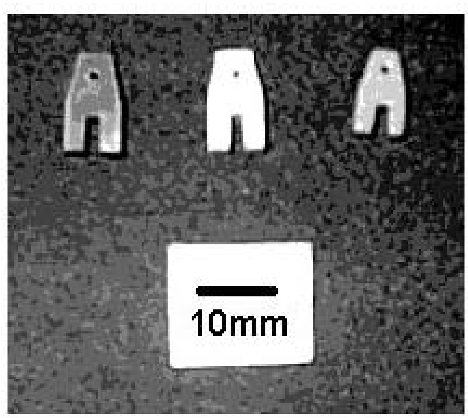

Figure 4 – Granite thread-guides

4. CONCLUSIONS

The potential for the use of granite residues as substitutes for more expensive, synthetic ceramics in the injection moulding process was demonstrated, and a series of operations was developed for that purpose. Successful binder removal was achieved without the introduction of macrodefects. In order to make the process more competitive, other binders and debinding methods are being developed. Preliminary sintering results have shown that the debound parts can be densified and can thus find commercial use in several industrial activities. Further studies are being conducted in order to assess and improve the final properties, thus broadening the range of potential applications.

Agradecimentos

The authors would like to acknowledge the financial support from CNPq and the help from Prof. Milton F. de Sousa (USP-São Carlos) and the Division of Mineral Technology of NUTEC-CE. The generous donation of materials by OPP Poliolefinas, Pierre Lira S.A. and IMARF was also appreciated.

REFERENCES

Beebas C. Mutsudy, 1991, Injection molding, Engineered Materials Handbook - Ceramics and Glasses, vol. 4, pp. 173 –180.

Brasileiro, F. E., 1999, Projeto resolitos – luxo que vem do lixo, Tecnologia & Inovação para a Indústria, Sebrae,

Braz, E., Amaral, M., A, 1996, A situação atual do setor de rochas ornamentais do Nordeste, Instituto Euvaldo Lodi.

Castelo Branco, S.M.A.,1998, Estudo do Resíduo de Rochas Ornamentais para Fins Tecnológicos, Projeto de Pesquisa para o CNPq.

Edirisinghe, M. J., 1991, Fabrication of engineering ceramics by injection molding, Ceramic Bulletin, vol. 70, n. 5, pp. 824 – 828.

German, R. M., 1989, Optimization of the powder-binder mixture for powder injection molding, Advances in powder metallurgy, vol. 3, pp. 51 – 66.

German, R. M, Bose, A., 1997, Ceramic injection moulding, Chapman & Hall, London, vol. 1 pp. 11-131.

Hwang, K.S., 1996, Fundamentals of debinding processes in powder injection molding, Reviews in Particulate Materials, vol. 4, pp. 71 – 104.

Lima Filho, V. X., 2000, Estudo da viabilidade técnica da substituição dos pós cerâmicos convencionais por pó de granito na injeção de peças cerâmicas à baixa pressão, Congresso Nacional de Engenharia Mecânica , Natal, RN.

Macéa, J. P., Macéa, J. R. , Nano, R. H., 1989, A aplicação de materiais cerâmicos na indústria têxtil, V Conferência Nacional de Tecnologia Têxtil e de Confecção, CETIQT, Rio de Janeiro, RJ.

Mangels, J. A., 1994, Low pressure injection molding, American Ceramic Society Bulletin, vol. 73, pp. 37 – 41.

Nogueira, R. E. F. Q. , 1992, Processing and properties of moulded ceramic bodies, PhD Thesis, Brunel University, UK.

Nogueira, R. E. F. Q., Bezerra A. C., Santos F. C., 1998, Produção de um guia-fio cerâmico por injeção a baixas pressões, V Congresso Norte-Nordeste de Engenharia Mecânica, Fortaleza, CE.

Relatório do NITES, Estudo geológico sobre mármores e granitos., Rochas de Qualidade , Edição 109, pp 78-81 de 1991.
Vidal, F. W. H., Bessa, M. F., Lima, M. A. B., 1999, Avaliação de rochas ornamentais do Ceará através de suas características tecnológicas, CETEM/MCT, Série Tecnologia Mineral, 74.