DESIGN SILICONE MOULDS FOR MANUFACTURING CERAMIC MICROCOMPONENTS

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RESUMO

O presente trabalho visa desenvolver micro moldes para o fabrico de microcomponentes de base cerâmica por uma técnica de processamento directa denominada gelcasting. Os modelos (madres) foram obtidos por estereolitografia/perfactory e posteriormente replicados em moldes de silicone de diferentes durezas Shore, de modo a seleccionar o que possuía qualidade superficial adequada ao fluxo das suspensões durante o enchimento, bem como fácil desmoldagem das peças em verde. Foram estudados diferentes tamanhos e geometrias, de forma a identificar as dificuldades e limitações dos diferentes pontos do processo.

ABSTRACT

This work pretends to develop micro molds for the manufacture of ceramic based microcomponents by a technical of direct processing called gelcasting. The models were obtained by stereolithography / perfactory and subsequently replicated in silicone molds of different Shore hardness, to select the proper flow of suspension for filling surface quality as well as easy demoulding of the specimens. Different sizes and geometries in order to identify the difficulties and limitations of the different points of the process were studied.

1. INTRODUCTION

Gel casting (GC) is one of the best performing techniques developed so far to fabricate complex-shaped ceramic bodies from high concentrated and low viscosity slurries that can be solidified by gelation once the mould is filled, thus facilitating mould removal prior drying and sintering. Although GC has been successful in producing both large to small size components, it is not suitable for the fabrication of micro-sized components (Yang et al., 2011, Young et al., 1991, Chan et al., 2000). However, recent results obtained by the authors (Olhero et al., 2012) opened the possibility to produce these microcomponents by casting a special ceramic formulation with enough green strength to withstand the high shear stresses generated upon demolding from the silicone molds.

This paper presents the results obtained in terms of silicone moulds and GC process variables. Manufacturing ceramic microcomponents by the GC process is a challenge task due to different factors inherent to the process. High solids concentration is demanded to promote enough green mechanical resistance for demolding operation and simultaneously reduce the shrinkage percentage, allowing a better dimensional control of the ceramic parts. On the other hand, the ceramic slurry should be sufficiently fluid to fill all mould cavities (Olhero, 2011). Considering that all mould features are micro size, it is common that air gets enclosure in the mould interior, generating undesirable defects. This way, before reaching the micro scale, one decided to test the process at the millimeter level.

Silicone is a flexible material that can be cured by different processes (condensation or addition), exhibiting different Shore hardness and shrinkage, covering applications like skin for prosthesis to more engineering applications, like moulds for wax injection of parts for investment casting of automotive industry components (Lino et al., 2004).

2. EXPERIMENTAL WORK

Preparation study of the silicone moulds

To test the ceramic GC process a silicone mould with millimetre features was produced. A simple E-shape was selected with three different dimensions by using an additive manufacturing stereolithography (SL) process. From these prototypes, moulds were manufactured with the silicone elastomer “SYLGARD(R) 184 from Dow Corning®, with a 50 Shore A hardness (see Fig. 1 for Shore hardness scale equivalences).

A ferroelectric ceramic slurry was prepared with 50 vol.% of BST ($Ba_{0.6}Sr_{0.4}TiO_3$) powder, in presence of 0.5 wt.% (based on the mass of solids) of dispersant (Dispex A40, Rivaz Química S.A, Portugal). Methacrylamide (MAM), methylenebisacrylamide (MBAM) and n-vinylpyrrolidinone (NVP) (all are AR Grade, Aldrich, Germany) were used as monomers in the gelcasting process. Tetramethylethylenediammine (TEMED) and ammonium persulfate (APS) (both are AR Grade, Germany) were used as the polymerization initiator and the catalyst, respectively. The mixture was cast by gravity into the silicone mould. The samples were left in the mould for 1 day at room temperature (although they could be demoulded after 1 h) and then were removed.

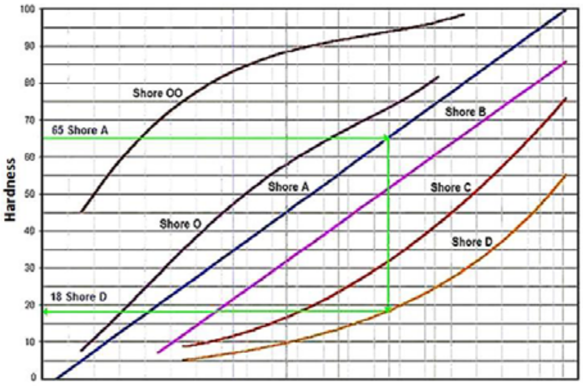

Fig.1- Equivalence among Shore hardness scales

After demolding, the samples were sintered following the cycle indicated in Fig. 2 (heating rate: 2ºC/min, dwell at 500ºC for 1 h, heating rate: 5ºC/min, dwell at 1350ºC for 4 h, cooling rate: 10ºC/min).

The surface roughness (Ra) of the stereolithography models was measured with the rugosimeter shown in Fig. 3, according to the directions presented in the Figure.

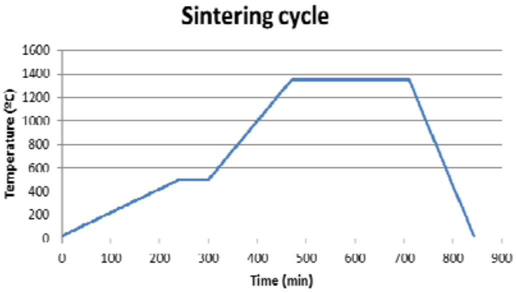

Fig.2 Sintering cycle

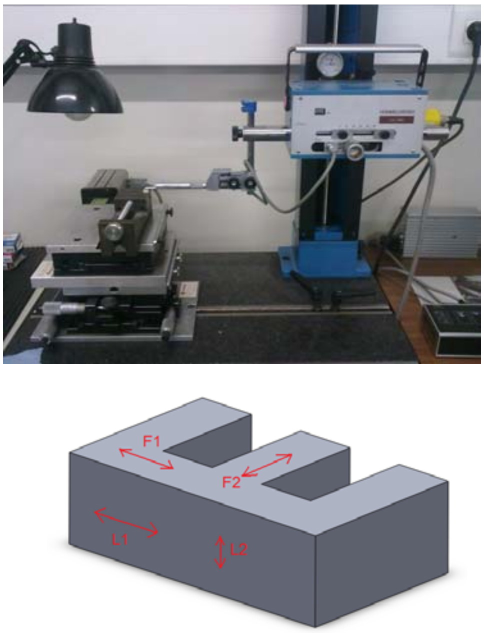

Fig.3 Rugosimeter Hommelwerke T800 and measurement arm TKL300

3. RESULTS

3.1 First silicone mould

Fig. 4 exhibits the three E-shaped prototypes (two of them with the same size but different drafts angles – 0 and 5°, see right side image) that in future experiments are going to be more reduced in size (micrometric scale).

Fig. 5 shows the silicone mould obtained from the stereolithography prototype with a detail of one zone. As one can see, there is some damage on the corners that have missing material and the extremes of the lateral bars show excess of silicone that escape underneath the stereolithography models. On the other hand, it was verified that the mould had low flexibility due to its exaggerated thickness (around 18 mm), which difficult the ceramic demoulding operation. Therefore, the thickness was reduced in the subsequent moulds manufacturing.

Fig. 6 presents two green (unfired) ceramic parts obtained by using this mould.

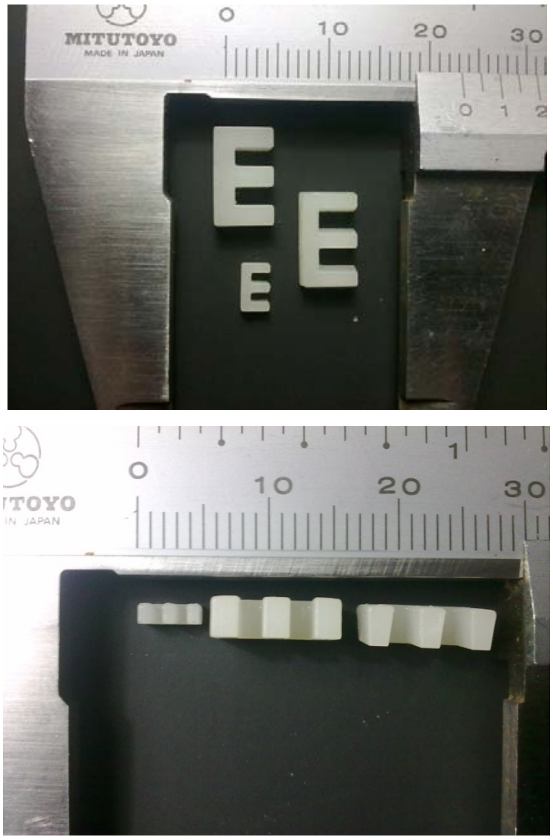

Fig.4 Stereolithography models for manufacturing the silicone mould

Fig.5 Silicone mould

This Figure shows that the parts have an undesired surface layer that is a result of ceramic particles segregation before the slurry hardening. The smaller particles tend to sediment and stick to the mould walls while the suspension is in a fluidic state. This defect has to be eliminated by improving the ceramic processing method, such as, reducing particle size and improving the rheological properties of the suspension.

After sintering, the ceramic parts exhibited good quality, no distortions and a controlled shrinkage; mean value around 19% (18.3% minimum and 22.5% maximum on the thinner sections).

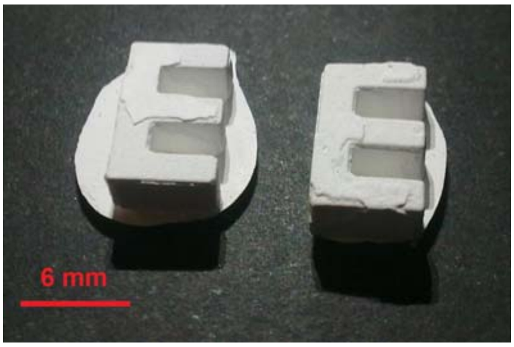

Fig.6 Green ceramic parts

The Ra values found vary from 1.1 to 1.9 µm (depending on the measurement direction).

The lateral wall roughness values are lower in vertical (L2) than in parallel direction to the base (L1), which is the opposite result of what was expected due to the layer effect of the AM processes. To understand this result, the topographic profile was obtained (Fig. 7). It can be seen higher peaks along the longitudinal profile (L1). The possible cause of these features, around 300µm peak-to-peak (L2) is the diameter of stereolithography laser. Considering that the prototyping model has reduced dimensions (the process is working close to the resolution limits), the effect of the laser beam diameter is transmitted to the topography of the parts. This result suggests finding alternative ways to make the models.

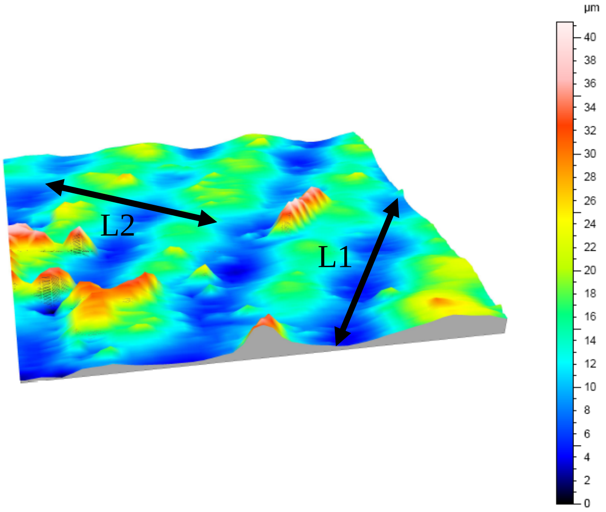

Fig.7 3D topographic analysis

The surface roughness of the ceramic components obtained shows values between 1.5 and 1.8 µm.

The results obtained in this first trial, gave important indications about the necessary changes to be introduced in all the stages of the manufacturing process to improve the final quality of the ceramic parts.

3.2 Second silicone mould

To avoid the silicone contact with the glue in the mould, which originated incorrect reproduction in some regions, the stereolithography models were made in the same plate (normal size and 50% reduction), as can be seen in Fig. 8. This plate can be glued to other base assuring that there are no interferences in the moulding region. Silicone moulds were manufactured with different Shore A hardness silicones; 40 (T4 from Dow Corning, USA), 30 (Dragon skin, Smooth-on, USA, or T4 from Dow Corning, USA, with 10% silicone oil) and 2 (Ecoflex from Smooth-on, USA) to test the feasibility of the demolding operation (see on Fig. 9 some of these moulds). The analysis of the moulds reveals that the lowest hardness (2 Shore A) enables an easy extraction of the ceramic parts, but the parts geometry is less exact.

Considering the resolution limitation of the stereolithography machine, smaller models were made using the Perfactory equipment of the EnvisionTEC (courtesy of the company, Germany). The size of the “E” letters was reduced 50 and 75% (see Fig. 10). This process promotes a surface roughness of 0.22µm, which is much lower than the models obtained by stereolithography (1.1-1.9 µm). Although this is a promising process to produce the prototypes, smaller dimensions could be necessary in the near future. First trials with INEGI new pulsed fibre laser (MOPA-M-HP-20 by Multiwave Photonics, USA), reveal that is possible to get uniform and isotropic material removal at a controlled

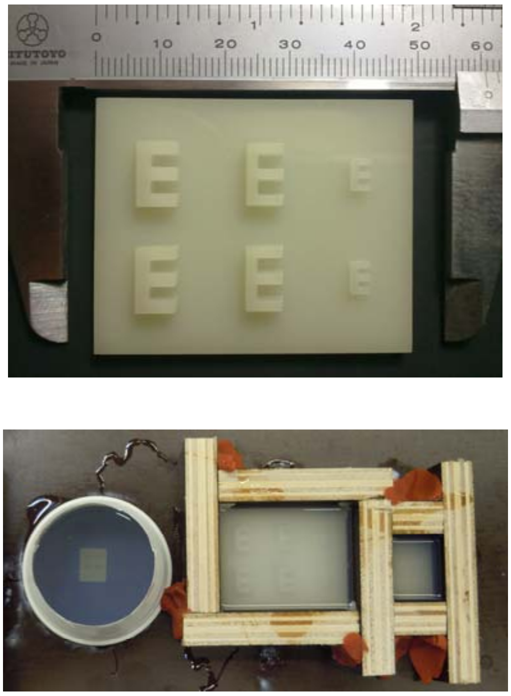

Fig.8 Stereolithography models made on the same plate and silicone moulds

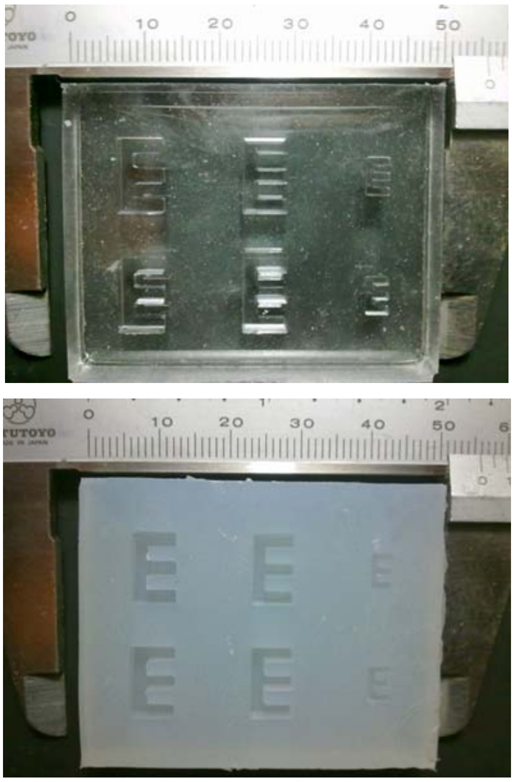

Fig.9 Silicone moulds; left: 40 Shore A, right: 2 Shore A

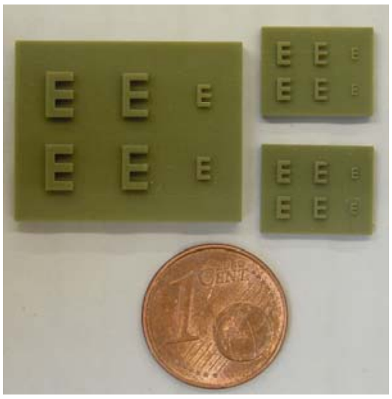

Fig.10 Perfactory models made on the same plate

surface depth, based on the inherent tight control of the laser-materials interaction and material transformation, producing narrows micro size cavities for casting the silicone moulds (Hendow et al., 2012).

The silicone moulds reproduced correctly all the models features, however, in the smaller models (reduced 75%, Perfactory process), some air bubbles were detected, as can be seen in Fig. 11.

The ability of the as obtained moulds to reproduce ferroelectric BST ceramic components was then tested. A well-dispersed suspension containing 50 vol.% solids with mean particle size of 0.5 μm and shear thinning behaviour was cast into the silicone rubber moulds. The E-shaped green bodies consolidated by gel casting are displayed in Fig. 12.

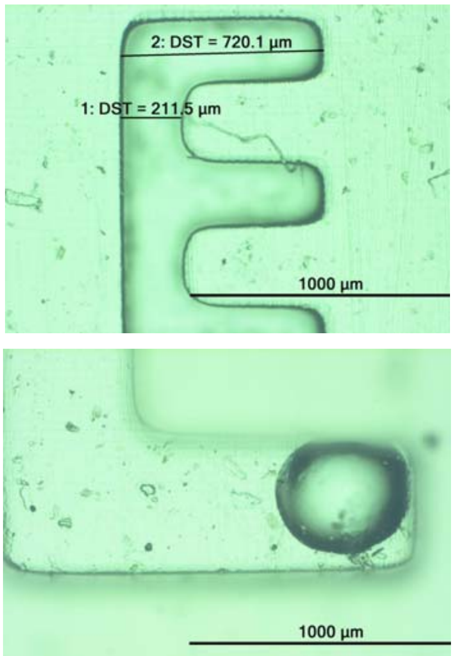

Fig.11 Silicone mould showing an air bubble

Fig.12. BST green samples obtained by gel casting process using the silicone moulds

In terms of dimensional limitation the process is conditioned by the difficulties in casting the ceramic slurry at room temperature. To overcome this problem external pressure should be used in the near future.

The use of external rectangular shapes instead of the circular ones used previously enhanced the flexibility of the mould and favoured the extraction of the ceramic samples from the mould. The mould thickness can also be reduced to facilitate the release of green bodies, which have to withstand torsion loads without tearing.

4. CONCLUSION

Ceramic microcomponents can be manufactured using silicone moulds and a gel casting process. Stereolithography models reveal to be adequate to produce small ceramic parts with adequate quality, however models with smaller sizes and higher resolution (lower surface roughness), such as the ones that can be obtained with the Perfactory process from EnvionTEC, can promote parts with better surface quality.

The analysis of the problems detected during all the manufacturing stages of the first models, gave insights to adopt some changes that were responsible for better reproduction of the models features.

Gravity casting of the ceramic slurry is possible for the cavity sizes tested. The first trials revealed a surface layer that is a result of sedimentation that demanded some changes on the ceramic processing. The changes introduced in the ceramic processing, such us, high efficiently powder milling corrected this problem.

When the parts start to be very small (microscale), ceramic vacuum casting is advisable.

The tests conducted until this moment were done with the ceramic parts extraction from the casting region, which introduces some limitations in terms of possible geometries, draft angles and surface finishing in the open region.

Future work will include improvements in the casting of the ceramic slurry into the silicone rubber moulds prepared from the Perfactory models and optimizing the process to avoid the presence of air bubbles.

If necessary, AM models can be substituted by models obtained by laser machining. If demoulding turns to be a problem, one can consider the manufacturing of silicone moulds with moving parts. Although more complex and more costly, these moulds can be cost effective in comparison with the conventional machining processes for producing ceramic microcomponents.

ACKNOWLEDGMENTS

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5. REFERENCES

Chan CM, Cao GZ, Stoebe TG. Net shape ceramic microcomponents by modified Sol-Gel casting. Microsystem Technology, 2000, 6, p. 200-204.

Hendow ST, Ozkan AM, Oliveira M, Mottay E. Pulsed fiber laser texturing of metals, silicon, and ceramics: comparison of surface structural changes induced by ns and fs pulses. SPIE Conf. Proc., Photonic West, 2012, paper 8243-26.

Lino FJ, Neto RJ, Paiva R, Moreira A. Rapid prototyping and rapid tooling in product development of ceramic components, Materials Science Forum, Advanced Materials Forum II, Vol. 455-456, TTT Publications, Proceedings of the II International Materials Symposium, April 14-16 2003, Caparica, Portugal, Edited by Rodrigo Martins, Elvira Fortunato, Isabel Ferreira and Carlos Dias, 2004, p. 835-38.

Young AC, Omatete O, Janney MA, Menchhofer PA. Gelcasting of Alumina. J. American Ceramic Society, 1991, 74, p. 612-618.

Olhero SM, Alves FL, Ferreira JMF. Last Advances in aqueous processing of aluminium nitride (AlN) – a review, Advances in Ceramics – Synthesis and characterization, Processing and Specific Applications, 2011, book 3, ch. 10, p. 207-242.

Olhero SM, Luis Garcia-Gancedo, Tim W Button, Fernando J. Alves, José MF Ferreira, “Innovative fabrication of PZT pillar arrays by a colloidal approach”, J. Eur. Cer. Soc., 2012, 32 (5), p. 1067-1075.

Yang J, Yu J, Huang Y. Recent developments in gelcasting. J. European Ceramic Society, 2011, 31 (14), p. 2569-2591.