

Preparation of ceramic microelectromechanical systems (MEMS) using a gelcasting consolidation technique

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Abstract

The present work focuses the preparation and characterisation of suspensions with three different powders, ferrite, PZT and alumina, for the fabrication of 3D-shaped ceramic structures by gelcasting. The characterization of powders by particle size is presented together with the preparation and characterization of the resulting suspensions by rheological and zeta potential measurements. Using 1-wt% of Dispex A-40 is possible to prepare concentrated PZT and ferrite suspensions to be consolidated by gelcasting. The workability of gelcasting technique is demonstrated on a range of component geometries suitable for MEMS applications.

Keywords: MEMS, colloidal processing, gelcasting, PZT, ferrites, alumina.

Introduction

Micro-electro mechanical systems (MEMS) are batch fabricated miniature systems with both electrical and non-electrical functionalities. Examples of such MEMS are inkjet print heads, miniature mechanical switches, acceleration sensors, etc...[1-2]. Currently, MEMS primarily use silicon technology and have typical feature sizes in the order of microns or smaller. However, in many practical applications, MEMS are too small to provide the required sensitivity as sensors or to provide the required forces as actuators owing to the small volume of active material [3]. Thus there has been considerable interest in the development of net shape, or near net shape processing routes in order to try to alleviate or minimise some of these problems.

Gelcasting is an attractive ceramic forming process for making high-quality ceramic parts [4-10]. Furthermore, it is also an affordable, rapid, and near net-shape forming process for producing complex shaped parts. In the process a high solids loading slurry obtained by dispersing the ceramic powders in the pre-mixed monomer and cross linker solution is cast in a mould of the desired shape. Adding initiator, monomer and cross linker polymerize to form a three-dimensional network structure, thus the slurry is solidified in situ and green bodies of the desired shapes with low polymer content are obtained. Gelcast green bodies have high homogeneity and mechanical strength, which is of great advantage for handling of the parts before sintering and for being able to produce large castings.

The most common materials in MEMS are silicon, metals and glass with more emphasis for silicon due to their excellent electronic, mechanical properties and the

well developed patterning and etching processes. However, many MEMS applications require properties that non-silicon compounds can offer. Piezoelectric materials like lead zirconate titanate (PZT) couple electric and mechanical energy. On the other hand, ferrite materials have both magnetic and dielectric properties. Accordingly, both classes of materials can serve as sensors and actuators in MEMS structures [11,12].

The present work aims to study the possibility to prepare 3-D shaped ceramic structures by gelcasting. For that, design of aqueous suspensions of two different materials, such as PZT and ferrite powders, suitable for gelcasting was the focal point. Since the preparation of high concentrated alumina suspensions is well established, the possibility to prepare samples by gelcasting is also tested and presented for comparison. Rheological behaviour and zeta potential were used to assess the degree of dispersion in suspensions. A range of different green shapes prepared with different materials has been demonstrated.

Experimental Procedure

Three different materials are used in the present work: PZT (TRS technologies, USA, D_{50} = 0.6 μm , surface area=5.106 m^2/g), ferrite (NZ50, Temex Ceramics, France D_{50} =2.45 μm , surface area=2.2391 m^2/g) and alumina (CT3000SG, Alcoa-Chemie GmbH, Ludwigshafen, Germany, D_{50} = 0.8 μm , surface area=7.0 m^2/g). The as-received Ferrite and PZT powders were ground by organic ball milling for 24h in a vibro energy mill and dried at 80C for 24h. Particle size distribution and specific surface area of the milled powders were determined by using laser particle size analyser and N_2 adsorption, respectively. The values of particle size and surface area presented above are related to the milled powders.

Zeta-potential measurements of all the powders studied were performed in the absence and in the presence of dispersant, using Coulter equipment (Delsa 440 Sx, Coulter, Buckinghamshire, UK). The powders were suspended into a background electrolyte solution of 1 mM KCl to insure a constant ionic strength. Each suspension was divided in two equal parts for increasing and decreasing pH runs, and the pH values were adjusted by using 0.1 M solutions of either NH_3 or HNO_3 , respectively.

Aqueous suspensions with 50-vol.% solids were prepared by dispersing the powders in an aqueous organic premix solution obtained by dissolving 20-wt% of methacrylamide (MAM, Sigma-Aldrich, Germany), N-N'-methylenebisacrylamide (MBAM, Sigma-Aldrich, Germany) and N-vinyl-2-pyrrolidone (NVP, Sigma-Aldrich, Germany) in 3:1:3 ratio in deionised water with the help of 1-wt% of dispex A 40 as a dispersing agent. The as-prepared suspensions were deagglomerated for 24h in a polyethylene bottle containing zirconia balls on a ball mill. The viscosity of the as-prepared suspension was measured using a rotational rheometer (TA Instruments, USA). The measuring configuration adopted was a cone and plate (4, 40 mm, gap 200 μm) and flow measurements were conducted between 0.1 and 800 s^{-1} . To evaluate dynamic properties and obtain information about the gelation behaviour in the linear viscoelastic region, time sweep with amplitude from 0 to 10 min at a constant frequency of 1 Hz was performed. The configuration adopted was the same used for flow measurements. The suspensions containing 50-vol% were consolidated by adding the polymerization initiator (10-wt% aqueous solution of ammonium persulfate, APS, Sigma-Aldrich, Germany) and the catalyst (tetraethylmethylenediamine, TEMED, Sigma-Aldrich, Germany) at the ratios of 4 and 2 $\mu\text{l}/\text{g}$ suspension, respectively. Afterwards, the suspensions were cast into non-

porous silicone moulds and were then allowed to set under ambient conditions until the completion of the gelling process.

Results and Discussion

Key factors for the successful production of ceramics by gelcasting are the homogenization and dispersion of the powder in the solution. Low solids loading and hard agglomerates in the suspension can result in deleterious surface and bulk defects in the final materials. Therefore it is necessary to produce a flowable slurry with at least 50 vol.% solids loading. The rheological behaviour of the ceramic suspension is determined by attractive van der Waals forces and repulsive electrostatic forces. Whereas the magnitude of the van der Waals forces are largely determined by the nature of the particles and the solvent, the repulsive forces can be modified over a wide range by adding dispersants. The zeta potential of the Ferrite and PZT particles without and in presence of 1-wt% of dispersant as a function of pH value is shown in Figure 1.

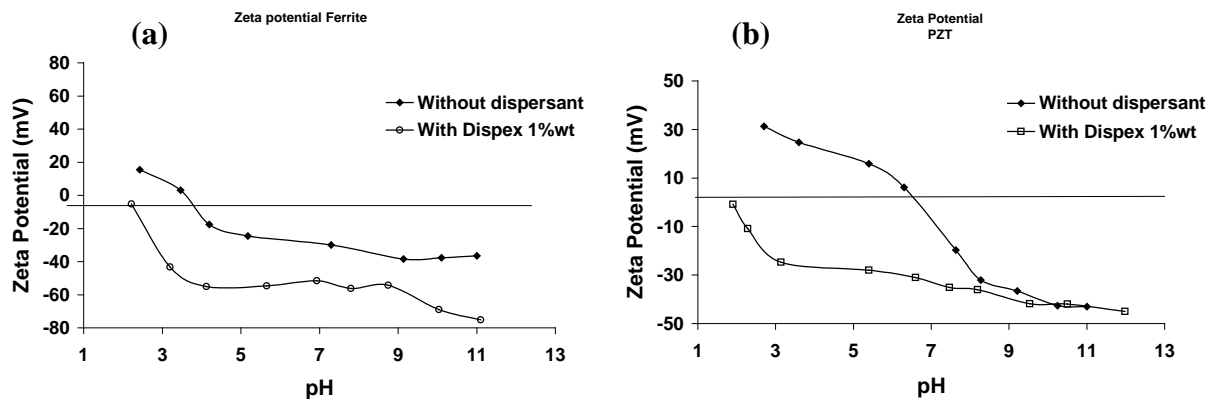


Figure 1. Zeta potential of (a) ferrite and (b) PZT powders in absence and in presence of 1-wt% of Dispex A-40.

The results show that the isoelectric point (IEP) of NZ50 ferrite powders is about pH=4, suggesting that acid pH environment are deleterious for dispersion. Adding 1-wt% of Dispex A-40, which is a solution of an ammonium salt of an acryl polymer in water, the IEP is moved to about pH=2 and in a wide pH range of 4-9, zeta potential value keeps almost constant. Contrarily to ferrite, IEP for PZT powders is at about pH=7, which means that neutral pH is disadvantageous for good dispersion and consequently for the preparation of high concentrated suspensions. Adding 1-wt% of dispex A-40, IEP is shifted for lower pH values and the particles present the highest zeta potential (around -50) at pH range 9-12.

In order to prove the dispersing ability of Dispex A-40, high concentrated suspensions were prepared and the resultant flow curves are presented in Figure 2. For comparison, rheological results for an alumina suspension with 50-vol.% is also presented. All the suspensions exhibited a shear thinning behaviour which is characteristic of well dispersed suspensions. When compared with PZT or alumina, ferrite suspension presents the highest viscosity values for all shear rate range tested, even with a lower solids loading. In fact, the highest solids loading allowed for ferrite suspension in presence of 1-wt% dispex A-40 is 45-vol.%. The difficulty to prepare high concentrated suspensions with ferrite powders could be related to the complex

surface chemistry which hinders deflocculation and consequently the increasing of solids loading.

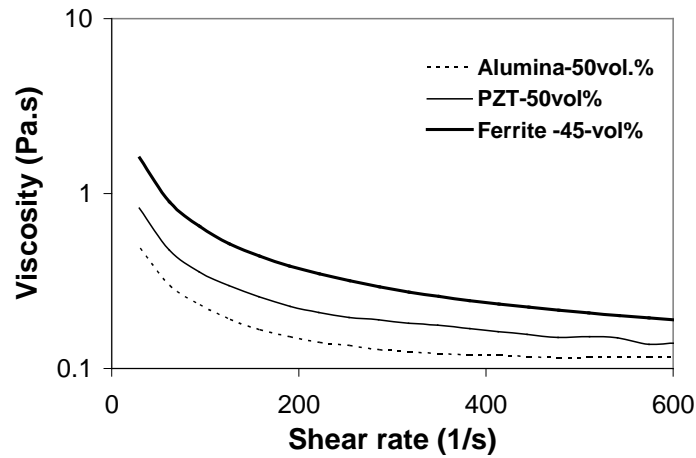


Figure 2. Flow curves of alumina, PZT and ferrite aqueous based suspensions with 50, 50 and 45-vol% solids loading, respectively in presence of 1-wt% dispex A-40.

The evolution of storage modulus, G' and the loss modulus, G'' of ferrite, PZT and alumina suspensions upon addition of polymerization initiator (APS) and catalyst (TEMED) as a function of reaction/gelation time are presented in Figure 3 (a-c), respectively.

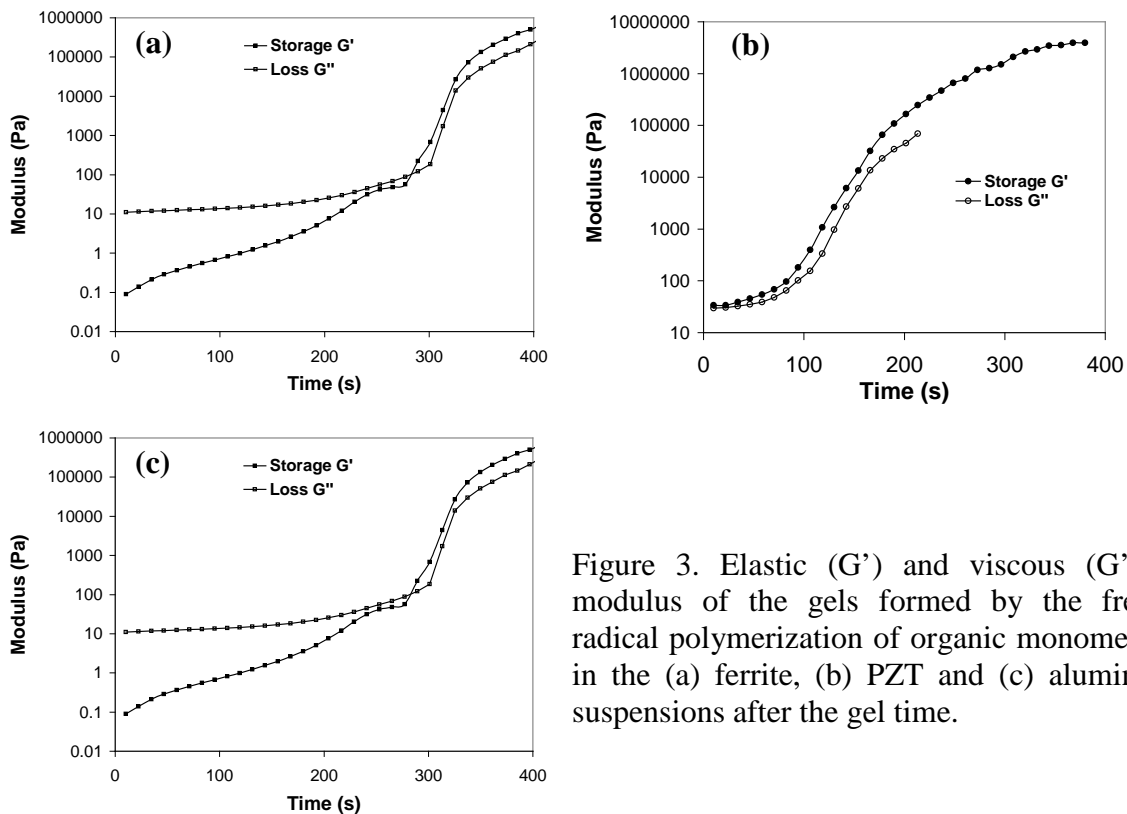


Figure 3. Elastic (G') and viscous (G'') modulus of the gels formed by the free radical polymerization of organic monomers in the (a) ferrite, (b) PZT and (c) alumina suspensions after the gel time.

The time at which the curves of G' and G'' intersect each other is taken as the gelation time, i.e., the time at which the elastic component, G' predominates over the viscous component. Both, the parameters G' and G'' increased with time increasing with the predominance of G' up to the crossover point. The crossover points occurred at the end of 1 min and 2 min for the PZT and alumina systems, respectively, and 4 min in the ferrite systems, meaning a higher time for gelation process of the ferrite slurry. Concerning the strength of the network structure after gelation time, ferrite system presents lower G' values when compared with alumina or PZT, which is indicative of a weak gel.

The applicability of these suspensions to prepare 3-D shaped green samples was also evaluated. Figure 4 illustrates different shapes of ferrite, PZT and alumina samples suitable for MEMS applications, obtained by gelcasting process.

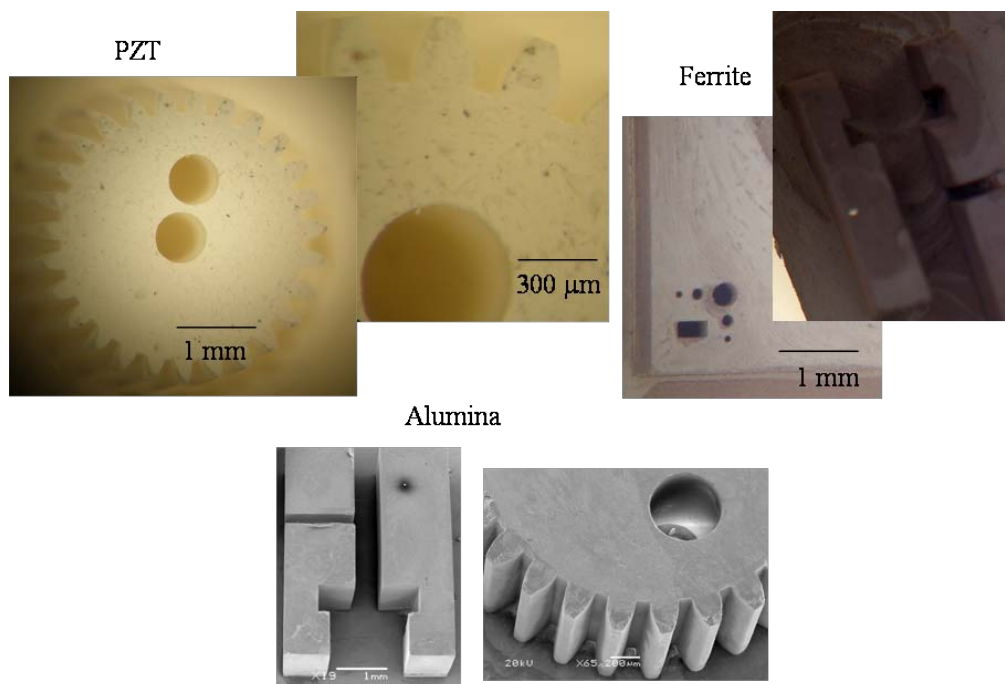


Figure 4. PZT, ferrite and alumina 3-D shaped samples obtained by gelcasting.

Conclusions

Ferrite and PZT powders have been used to prepare high concentrated suspensions in aqueous media using an ammonium polyelectrolyte as dispersant. These suspensions were successfully used for near net shaping of ceramic parts by gelcasting, a process that enables minimisation of the expensive post sintering machining operations, therefore decreasing the production costs of ceramic components.

Acknowledgments

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