Forming Alumina Parts Using Acrylamide Gels

Abolfazl Barati¹, Mehrdad Kokabi*, and Mohamad Hossein Navid Famili

Polymer Engineering Group, Tarbiat Modaress University (TMU), P.O. Box: 14115-111, Tehran, I.R. Iran Received 20 July 2002; accepted 14 December 2002

ABSTRACT

limited to the processes required extensive and expensive machining (i.e., injection moulding or pressure slip-casting). These technologies for shaping ceramics, however, possess several shortcomings that limit their use as a complex-shape forming method. Because of these limitations, "gel-casting" which was lacking in above deficiencies, has been proposed as a general and desired method for shaping any ceramic powder. In this work, gel-casting of α -alumina is investigated based on in situ polymerization and gelation of acrylamide monomers as the setting mechanism for forming the green part. Special attention was paid to the preparation of fluid, castable slurries with a high loading level of α -Al₂O₄ (>80 wt.%) and low viscosity (0.65 Pa.s) by using polyelectrolyte dispersants (i.e., poly(methacrylic acid) salts), also gelation of acrylamide (effect of gelation factors on idle time), mould materials, drying of parts through the liquid desiccant drying method and its comparison with conventional drying technique, green machining, debinding and sintering.

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Key Words:

gel-casting; α-alumina; acrylamide; drying; green machining; de-binding; sintering.

INTRODUCTION

Ceramic parts are formed by consolidating powders into the desired and often rather complex shapes and densifying them at higher temperatures. The main challenge in this process is to achieve parts with defect-free microstructures because pores, inclusions, or any other inhomogeneity of the microstructure cause stress concentration during loading of a part, and subsequently the risk of failure. As more advanced ceramics are being used in industry [1], it is a great challenge to fabricate such complex-

(*)To whom correspondence should be addressed. E-mail: mehrir@modares.ac.ir shaped parts with high reliability at acceptable costs. An ongoing focus is the development of near-net shape fabrication processes that can produce complex-shaped parts with a minimum of machining by optimizing and controlling each fabrication process step in order to minimize the number and size of microstructural defects to within design limits [2].

The existing forming processes, such as slip-casting, pressing and injection moulding possess several shortcomings that limit their use as a complex-shape forming methods and do not conform in all of the aforementioned requirements [3-5].

As such, forming of the parts by using organic gels, gel-casting is a net-shape forming process similar to the other near-net shape forming processes (i.e., pressure slip-casting, injection moulding), but with several advantages [6]. Compared to slip-casting, gel-casting results in much more homogeneous material with no density differences over the parts. Compared to injection moulding, gel-casting does not need expensive moulds, uses only small quantities of organic binder (typically 4 wt.%), uses water or other low viscosity solvent as the suspension media and does not include the critical binder removal step [7].

The advantages of gel-casting can be summarized

as, (a) capability of producing complex parts, (b) ease of implementation owing to its similarity with other suspension forming technique, (c) low equipment cost, (d) low cost mould material, (e) high green strength, (f) excellent green machinability, (g) very homogeneous material properties and (h) ease of binder removal compared to injection moulding [7].

Other techniques like freeze-casting [8], aqueous injection moulding [9], direct coagulation-casting (DCC) [10] and hydrolysis-assisted solidification [11] are gaining renewed attention by the success of gelcasting.

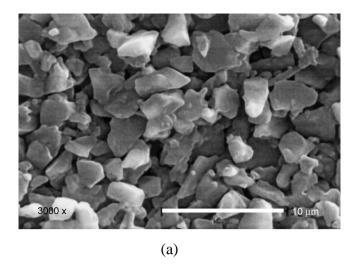
In our group the development of gel-casting, based on the research results published by the Young et al. [12] started 10 years ago, but the first article on gel-casting of engineering ceramics, mainly reaction bonded silicon, was published a few years later [13]. In this context, the aim of this paper is to investigate the gel-casting of Al₂O₃ powders; preparation of fluid, castable slurries with a high Al₂O₃ loading level, gelation of acrylamide as a polymeric binder and the parameters effect on it, choosing the mould material, drying of parts through the liquid desiccant drying method and comparing it with conventional drying techniques, green machining, de-binding and sintering.

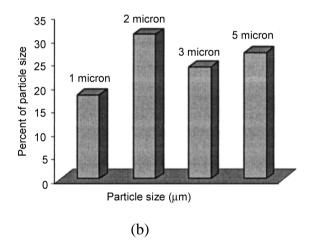
Table 1. Composition of samples.

Material	Function	Chemical formula	Characteristics	Supplier	
Fused α-alumina	Ceramic powder	Al ₂ O ₃	Figure 1 (a to c)	Good Fellow ^a	
Acrylamide (AAm)	Monofunctional monomer	C ₂ H ₃ CONH ₂	M _w 71.08 Soild white powder	Merck ^b	
N,N'-Methylene bis acrylamide (MBAM)	Bifunctional monomer (cross-linker)	(C ₂ H ₃ CONH ₂) ₂ CH ₂	M _W 154.2 Solid white powder	Sigma ^c	
Ammonium persulphate (APS)	Initiator	(NH ₄) ₂ S ₂ O ₈	M _W 228.2 Solid white powder	Merck ^b	
N,N,N',N'-Tetra methylethyl diamine (TEMED)	Accelerator (catalyst)	C ₆ H ₁₆ N ₂	M _W 116.2 Liquid, yellow	Merck ^b	
Poly(ethylene glycol) (PEG)	Liquid desiccant	HO(C ₂ H ₄ O) _n H	Ave. M _W 950-1050	Merck ^b	
Sodium salt of poly(methacrylic acid)	Dispersant	(C ₄ H ₅ O ₂ Na ⁺) _n	Ave. \overline{M}_{W} 15000 Aqueous solution 40 wt.%	R.T. Vanderbilt ^d	
Ammonium salt of poly(methacrylic acid)	Dispersant	(C ₄ H ₅ O ₂ -N ⁺ H ₄) _n	Ave. \overline{M}_{W} 15000 Aqueous solution 40 wt %	R.T. Vanderbilt ^d	

⁽a) Cambridge Science Park, Cambrige CB44DJ, England; (b) E. Merck, D6100 Darmstadt, Germany;

⁽c) Sigma Chem. Co, USA; (d) R.T. Vanderbilt Co., USA. (Figure 1)





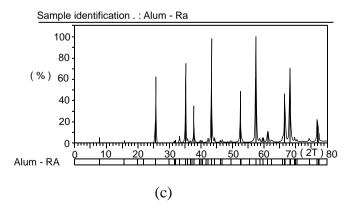


Figure 1. (a) Shape, (b) particle size distribution and (c) x-ray diffraction of α -alumina powder.

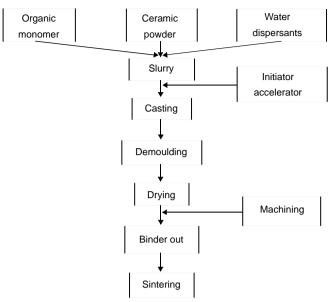


Figure 2. General gel-casting process flowchart.

EXPERIMENTAL

Materials

The specification of the materials used in this work are summarized in Table 1 and Figure 1.

Instruments

Instruments which were used in this study are as follows:

- Brookfield viscometer (Shanghai balance instrument factory, model NDJ-4, type LVF).
 - One axis tools machine (Tabriz, T N50B_R).
- Scanning electron microscopy (Philips XL30, magnifying to 10^{5}).
- Programmable furnace with computer controlling, (Exciton, model Azar 1500).

Table 2. Composition and viscosity of aqueous mixtures of binders (pre-mixes).

Pre-mix	AAm (wt %)	MBAM (wt %)	AAm / MBAM	Viscosity (mPa.s)
A1	5	0.4	12.5	0.9
A2	6	0.2	30	0.9
А3	7	0.2	35	1.0
A4	8	0.2	40	1.0
A5	9	0.2	45	1.1
A6	10	0.2	50	1.2

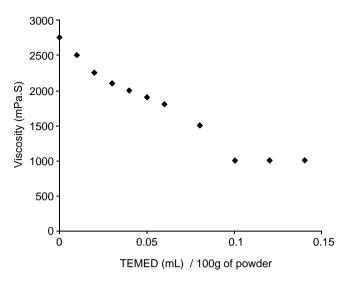


Figure 3. Effect of TEMED as dispersant on the viscosity of 35 vol% alumina slurry.

- Ultrasonic shaker (Bransonic model B200, output frequency: 40 kHz).
 - pH meter (Eutech instruments, type WP2).

Procedure

Slurry Preparation and Casting

The gel-cast process flowchart typically looks like the other ceramic processes especially slip-casting, (2) [12]. Firstly, by using magnetic or mechanical stirrer (with turbine blade), pre-mix solution that contains monomers, solvent and dispersant is prepared. The composition of the pre-mixes and their viscosity are given in Table 2. After adding the dispersant to the pre-

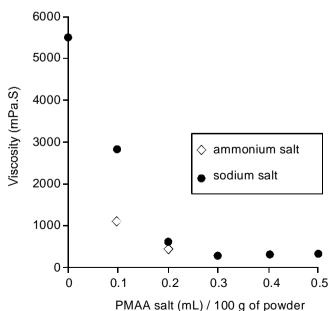


Figure 4. Effect of ammonia and sodium salts of PMAA on the viscosity of 45 vol % alumina slurry.

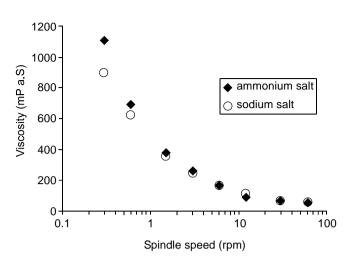


Figure 5. Comparison between viscosity reduction of polyelectrolytes on 40 vol% alumina slurry in 0.3 mL of polyelectrolyte per 100 g of powder.

mix solution, alumina powders with various loading levels were added to solution and stirred by using mechanical stirrer for at least 2 h to promote dispersion.

The resulting slurry was degassed by ultrasonic shaker. Vacuum was also applied until no further air bubble was released. Then accelerator was added and gently agitated for at least 15 min. A 10 wt% aqueous solution of ammonium persulphate, i.e. the initiator, was added to slurry and manually stirred for 1 min. After that the slurry was cast into the mould at room temperature. A combination of epoxy and steel moulds were used in this work. A non-silicone based mould release agent (dioctyl phthalate) was also employed. Gelation took place in 3 to 75 min after casting, depending on the processing conditions. After completion of polymerization process, the ceramic green part was demoulded and dried through liquid desiccant

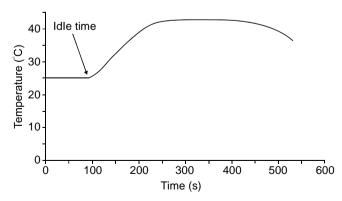


Figure 6. Change of pre-mix temperature during gelation (typical pre-mix which has 7.5 vol% of acrylamide and molar ratio of AAm per MBAM equal to 10).



Table 3. Experimental design for determining the effects of various factors on the idle time of gelation process.

Exp. no	Monomer concentra- tion (wt %)	Molar ratio of monomer to cross-linker	Molar ratio of initiator to total monomers	Temperature (°C)	Ceramic loading level (vol %)
1	6	22	0.2	25	0
2	7	22	0.2	25	0
3	8	22	0.2	25	0
4	9	22	0.2	25	0
5	6	16	0.2	25	0
6	6	18	0.2	25	0
7	6	20	0.2	25	0
8	6	22	0.2	25	0
9	6	22	0.2	25	0
10	6	22	0.3	25	0
11	6	22	0.4	25	0
12	6	22	0.5	25	0
13	6	22	0.2	15	0
14	6	22	0.2	25	0
15	6	22	0.2	35	0
16	6	22	0.2	45	0
17	6	22	0.2	25	20
18	6	22	0.2	25	30
19	6	22	0.2	25	35
20	6	22	0.2	25	40

method by immersing it into 20 wt% of aqueous or non-aqueous solution of PEG1000. After 4 h, green part, which lost about 25-30 wt% of its water content, was removed from liquid desiccant and drying followed in an oven at 60-80°C. Once dried, the gel-cast green bodies are sufficiently strong for machining operations. After machining, polymer was burnt out under controlled heating program, which was deduced from thermogravimetric analysis (STA) of acrylamide gels. Subsequently sintering of the part was carried out in air. A typical sintering scheme was 0.1 C/min to 600 C and 2 C/min from 600 to 1450 C.

Measuring of Physical and Chemical Properties

Particle size and particle size distribution of alumina powder were measured by scanning electron microscopy (Figure 1). Viscosity of pre-mix solution and slurry with and without dispersing agent were measured by Brookfield type viscometer, using spindle No. 1 (for pre-mix) and No. 2 (for slurry) at angular velocity of 3 rpm in water bath at 25 C. The variations of viscosity versus shear rate were monitored. All

measurements were repeated to check for reproducibility and averaged.

The bulk densities of complex-shaped gel-cast parts after sintering were determined by Archimedes method.

RESULTS AND DISCUSSION

Solid Loading of the Slurry

In gel-casting, the slurry is cast in a non-porous mould wherein it is subsequently set by the gelation reaction. Accordingly, the green density of the part formed corresponds to the solid loading of the slurry. Hence, it is beneficial to obtain a solid loading as high as possible in order to achieve higher dimensional tolerances by minimizing the possible distortion during drying and sintering shrinkage. On the other hand, the shaping of intricate parts requires the slurry to easily fill moulds of possible complex geometry. One way of achieving this is to use pre-mix solution of low viscosity. As shown in Table 2, the pre-mixes A1 to A6 (which contain 5 to 10

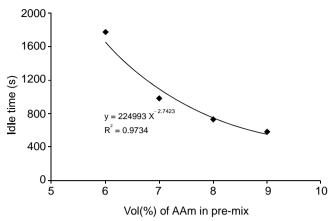


Figure 7. Effect of acrylamide concentration on idle time of gelation of pre-mix solution.

wt % binder) have viscosities similar to that of pure water (i.e., 0.9 mPa.s).

Using a proper dispersant for the powder is also essential to prevent any participation in slurry. Here, three dispersants were tested. These included sodium and ammonium salts of poly (methacrylic acid) and TEMED. TEMED, Which is used as an accelerator in gelation process, acts as a good dispersant for our powder. TEMED With two amine groups is adsorbed on the surface of the powder, prevents strong hydrogen bonding between water and alumina by making poor hydrogen bonding with water.

As shown in Figure 3, viscosity of the slurry was significantly decreased by adding small amount of TEMED. Concentration of TEMED in slurry must be carefully controlled until gelation would not occur during casting in a mould. Sodium and ammonium salts of poly (methacrylic acid), which act as polyelectrolytes, are adsorbed on ceramic powder surface and prevents their coagulation [14]. As shown in Figure 4, by using these polyelectrolytes, viscosity of the slurry was high-

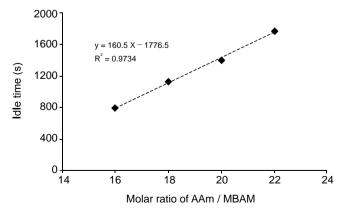


Figure 8. Effect of acrylamide concentration on idle time of gelation of pre-mix solution.

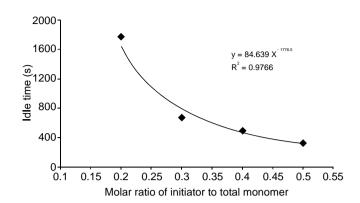


Figure 9. Effect of initiator concentration on idle time of gelation of pre-mix solution.

ly decreased (21 times). The minimum viscosity is obtained at about 0.3 mL of both salts per 100 g of dry powder. Higher concentration of polyelectrolytes results in an increase in the slurry's viscosity. As shown in Figures 3 and 4, addition amount of dispersants to slurry has a limiting value. At this level and above it, the particle surface is saturated with the adsorbed dispersant and that the viscosity of solution is no longer dominated by the concentration of dispersant. Also, excess amount of polyelectrolyte in solution results in free polymer, which increases the viscosity of slurry. Figure 5, compares performance of the polyelectrolytes on viscosity reduction of alumina slurry. The sodium salt was better than the others but in the processing of advanced ceramics, ammonium salt is commonly preferred because the sodium salt can lead to the formation of undesirable liquid phase during sintering.

Gelation Process

In this study, polymerization occurs under free-radical mechanism, which is an exothermic reaction. Thus, the

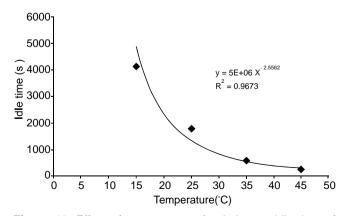


Figure 10. Effect of temperature of solution on idle time of gelation of pre-mix solution.

Table 4. Results of gel-cast parts green machining.

Exp.	Type of ceramic	Average particle size (μm)	Monomer concentration (vol %)	Molar ratio of monomer to cross-linker	Weight ratio of plas- ticizer to monomers	Angular velocity of spindle	Forwarding velocity of the tools on the part (feeding rate)	Water content of the part (wt %)	Turnings morphology	Edge and corner conditions	Surface finish
1	Alumina	30	5	40	None	180	Low	0	Dust	Small chips	Good
2	Alumina	30	5	20	None	180	Low	0	Dust	moderate	Good
										chips	
3	Alumina	4	5	40	None	180	Low	0	Small shavings	Small chips	Good
4	Alumina	4	7.5	40	None	180	Low	0	Very Small	Very small	Good
									shavings	chips	
5	Alumina	4	5	40	Glycerol (10%)	180	Low	0	Mostly dust	Small chips	Good
6	Alumina	4	5	40	Glycerol (20%)	180	Low	0	Small shavings	No chips	Excellent
7	Alumina	4	5	40	Glycerol (30%)	180	Low	0	Large shavings	Plastic deformation	Excellent
8	Alumina	4	5	40	Glycerol (10%)	180	Low	5	Small shavings	No chips	Excellent
9	Alumina	4	5	40	Glycerol (10%)	180	Low	10	Large shavings	Plastic deformation	Excellent
10	Alumina	4	5	40	Glycerol (20%)	325	Low	0	Dust	Moderate chips	Excellent
11	Alumina	4	5	40	Glycerol (20%)	180	High	0	Large shavings	Huge chips	Bad
12	Silicon	5	5	40	Glycerol (20%)	180	Low	0	Small shavings	No chips	Excellent

initiation of polymerization in pre-mix can be determined by changes in the temperature of solution. The idle time, t_{idle} , i.e. the time between the addition of the initiator and the beginning of polymerization can be monitored. This is equivalent to the time available for casting the slurry during processing. Figure 6, shows the changes in temperature of a pre-mix solution during gelation and indicates the idle time.

Idle time is affected by the factors such as monomer concentration, monomer to cross-linker ratio, initiator concentration, amount of ceramic loading level in slurry and solution temperature, (Table 3). Figures 7-11 show the variation of idle time with any of the above factors. As shown in those Figures, idle time decreases with increase of monomer concentration, initiator concentration, amount of ceramic loading level

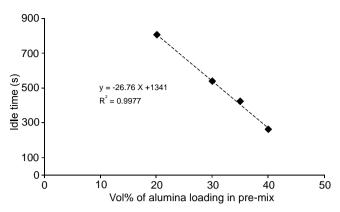


Figure 11. Effect of ceramic loading level on idle time of gelation of pre-mix solution.

and solution temperature, but increases with increase of monomer to cross-linker ratio. Effect of monomer to cross-linker ratio and amount of ceramic loading level on idle time, in the range of experimental conditions, are linear but the other factor affects exponentially. Although, the effect of the solid filler in accelerating the gelation (curing) reaction was reported for thermoset polymers [15-17], but for as-yet undetermined reasons, for catalytic role of ceramic powder, the idle time is reduced significantly.

Casting Moulds

Gel-casting moulds can be fabricated from a wide range of material including metals, glass, petroleum wax and polymeric materials. Since the casting slurries present low viscosities, the mould needs to be designed in order to avoid any leakage. Gel-cast parts are weaker than injection moulded part. Hence, the mould should allow to be removed from the parts (vs. the part being removed from the mould). In order to make

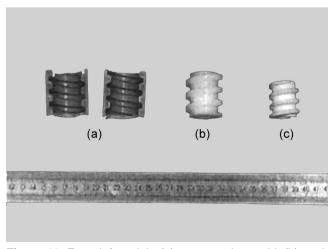


Figure 12. From left to right (a) epoxy resin mould, (b) positive model of the part and (c) sintered part.

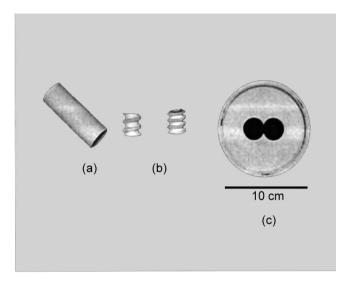


Figure 13. Left to right (a) tube, (b) screw and (c) machined complex alumina parts formed by gelation of acrylamide.

unmoulding easier, effective mould release agents are needed (e.g., dioctyl phthalate (DOP), silicon-based spray or oil, non-silicon based oil, etc.). Some mould release agents will also inhibit the polymerization reaction (e.g., silicon-based spray or oil).

With this in mind, we have investigated the use of several mould materials. The compatibility with the gelation reaction and ease of unmoulding has been assessed qualitatively. We have used different mould materials as metal, Plexiglas and casting epoxy resins. The latter is interesting, as it is cheap and can be used to fabricate a complex mould starting from, e.g. a pos-

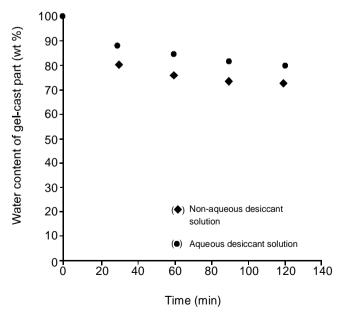


Figure 14. Drying of alumina cylinder (9 mm diameter with L/D = 4) in aqueous and non-aqueous solution of PEG1000.

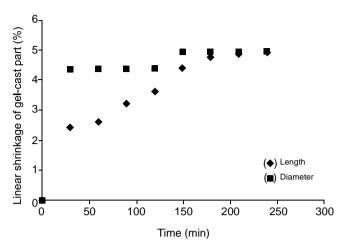


Figure 15. Linear shrinkage of alumina cylinder in aqueous solution of PEG1000.

itive of the part (Figure 12).

Forming

As mentioned earlier, the fabrication of complexshaped ceramic parts has been limited to processes requiring extensive and expensive machining; also these existing forming processes have inherent problems. For example, one of which is the fabrication of parts containing thick and thin sections. In this study, many simple and complicated parts have been formed by gel-casting. Figure 13 shows some parts produced in this work, which indicates the capability of this process for forming both simple and complex shapes.

Drying

Conventional methods in drying ceramic parts, using dry air or air with particular conditions of humidity and

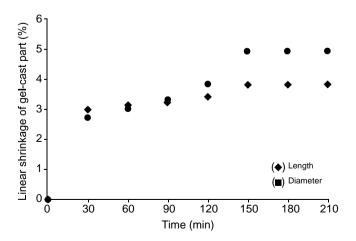


Figure 16. Linear shrinkage of alumina cylinder in non-aqueous solution of PEG1000.

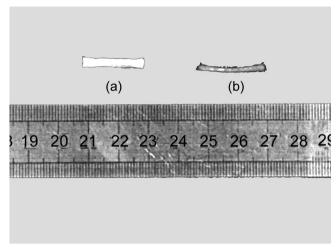


Figure 17. From left to right (a) acrylamide pure gel-dried in non-aqueous solution of PEG1000, (b) same gel dried in air at room condition.

temperature usually confront with many difficulties. Non-uniform and differential drying in various regions due to the solvent gradient, induces structural and residual stresses. They cause defects such as cracking, warping, bending and other malformations, making the parts useless. In this work, a novel method has been developed for drying gel-cast ceramic parts, which not only increased the drying rate but also removed all of the defects due to the release of residual stresses [18].

In this method, by using liquid desiccant (aqueous or alcohol solution of PEG 1000), the parts loose safely about 20-30 wt% of interior solvent (water) in a very short time, i.e. 2 h, (Figure 14). It is really a breakthrough in this critical stage of the process. The linear shrinkage of the part is nearly uniform (Figures 15 and 16). The liquid desiccant can provide buoyant support for the article being dried. Such support can be helpful

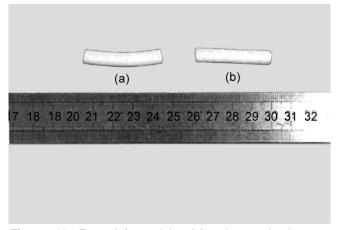


Figure 18. From left to right, (a) gel-cast alumina part (35 vol %) dried in room condition, (b) same part dried in aqueous solution of PEG1000.

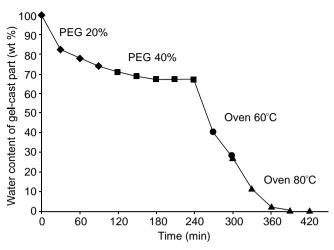


Figure 19. Drying of alumina gel-cast part in various concentration of non-aqueous solution of PEG1000 and in oven at 60 and 80°C.

in preventing distortion of the article during drying (Figures 17 and 18). After the part passes the critical stage of drying safely, it can continue to dry in the air at room condition or in an oven at higher temperature without any danger (Figure 19).

Machining

Machining of a green part is a cost effective technique since the method does not require special hard tools as for the machining of sintered ceramics. The machinability of a green parts depends on various factors such as monomer concentration, monomer to cross-linker ratio, average particle size, rotation velocity of part around the axis of machine (rotational velocity of spindle), forwarding velocity of tools on the part (feeding

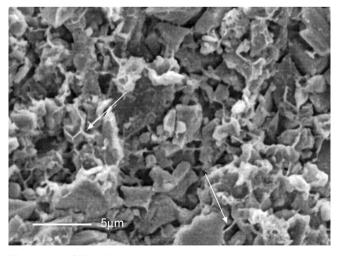
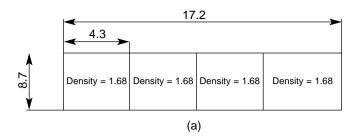


Figure 20. SEM Photograph of green alumina gel-cast part (50 vol.% loading in slurry). Note the binder networks holding the alumina particles together, as indicated by arrows.



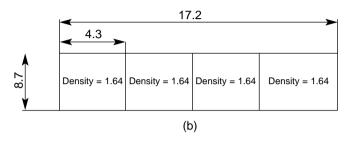


Figure 21. Comparison of dimension and density in (a) cylindrical alumina part dried in aqueous liquid desiccant solution (20 wt %) and in oven at 100°C, (b) after debinding at 600°C and rate of 2°C/min.

rate), water content of the part and amount of plasticizer which was added to the slurry before casting. Based on the results of Janney et al. [19], experiments were designed as in the following details.

As shown in Table 4, lower concentration of monomer and monomer to cross-linker ratio cause larger chips on the edge and corner of part during machining. This defect can be minimized by using plasticizer or machining the wet green part (with small water content therein). The amount of plasticizer or water content

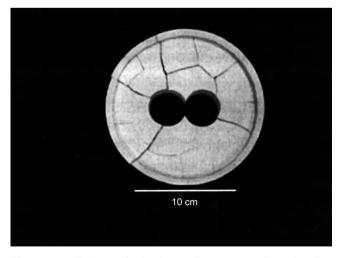


Figure 22. Failure of alumina gel-cast part after sintering at 1450°C because of knit lines therein.

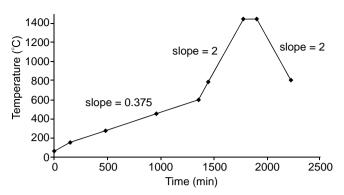


Figure 23. Typical sintering scheme.

of part has a limiting value. Above this level, plastic deformation occurred on the corner or edge of the part. Higher rotational velocity of spindle and feeding rate, increased the chips on the corner and edge of the specimen part. Type of ceramic had no effect on the machining conditions of the green parts.

Debinding

As mentioned earlier, in the gel-casting system ceramic particles bind together by using polymeric network (Figure 20). For achieving higher density, this polymeric binder should be removed from the green part, i.e. oxidation at higher temperature. The pre-mix solutions, which are used in this work, contain a maximum 10 vol.% polymeric binder (Table 2). As a result, the dry green parts contain only 4 wt% binder that is often lower than the binder content in commercial powders for compaction. Unlike polymer- or wax-based injection moulding, binder removal is neither time consuming, nor critical. STA Analysis of pure acrylamide gels indicates that oxidation of the acrylamide binder starts at 350-400 C [20]. Above this temperature the heating

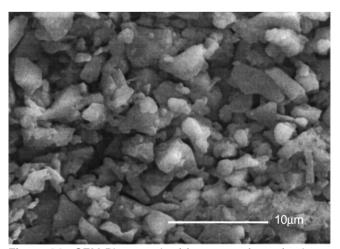


Figure 24. SEM Photograph of fracture surface of gel-cast alumina part sintered at 1450°C.

rate has to be carefully controlled to avoid build up of excess internal pressure (i.e., 0.1 C/min). As seen in Figure 21, there is no shrinkage in the part during debinding.

Sintering

After sintering, knit lines and air bubbles, which are the major defects, were found in the gel-cast parts [6]. The knit lines were formed during the gelation stage because the curvature and orientation of lines suggested them (Figure 22). This defect was not observed when slurry was gently agitated after casting and before gelation, and also when long idle time was used.

The other defect, inherent to solvent-based processes, is entrapped air bubbles, which is tried to be removed by using an ultrasonic shaker. A typical sintering scheme was shown in Figure 23. Figure 24 shows the SEM photograph of fracture surface of gelcast alumina part sintered at 1450 C.

The densities of sintered alumina parts were measured by Archimedes method (Table 5). All the measurements are repeated to check for reproducibility and averaging. During sintering the shrinkage of the part was isotropic and averaged 8% to 9% linearly for a slurry containing 40 vol % of the alumina powders. These results indicate that the gel-casting is a near-net shape forming technology with predictable dimension.

CONCLUSION

A generic near-net shape forming process based on the in-situ polymerization and gelation of acrylamide monomer binder, has been evaluated using commercial alumina powders with specific characteristics. The process was found to have the following features:

- (i) The alumina slurry has low viscosity at high solids loading (50 vol.% or >80 wt %) by the aid of polyelectrolytes and TEMED as an accelerator in polymerization reaction.
- (ii) The forming process is capable of producing near-

Table 5. Results of Archimedes test for alumina parts after sintering at 1450°C.

Characteristics	Fired at 1450°C	Fired at 1450°C		
Onaracionstics	and holder for 1 h	and holder for 2 h		
Bulk density (g/cm ³)	2.4732	2.6077		
Open porosity (%)	28.37	27.72		
Relative density (%)	62.14	65.54		
Close porosity (%)	9.49	6.74		

net complex shapes.

- (iii) The time available for casting the slurry (idle time) can be controlled by the amounts of initiator and catalyst added to the slurry as well as by the composition and processing condition such as monomer concentration, monomer to cross-linker ratio, ceramic loading level and temperature.
- (iv) By using liquid desiccant drying method, the parts lose safely about 20-30 wt.% of interior solvent (water) in a very short time without creating any defect or deformation in them.
- (v) The green dried gel-cast parts are sufficiently strong for machining operation. The machinability of a green part depends on factors such as monomer concentration, monomer to cross-linker ratio, average particle size, rotation velocity of the part around the axis of machine, forwarding velocity of the tools on the part, water content of the part and amount of plasticizer.
- (vi) The binder concentration is low, less than 4 wt % of the dried solid. Therefore, binder removal is neither time consuming, nor critical.
- (vii) The total linear shrinkage of a 40 vol % alumina slurry was 4% during drying and 8% to 9 % during partial sintering.

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