Pressure Slip Cast Processing of Alumina (Al2O3) Products and Comparative Evaluation of Mechanical Properties

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Abstract

Pressure Slip Casting (PSC) using polymer molds offers several advantages over Conventional Slip Casting (CSC) of ceramics such as enhanced productivity in combination with higher green density, homogeneity and low rejections. PSC is currently practiced in table-ware industries however, application to the technical ceramics is limited due to the collapse of cast part while de-molding during pressure cast cycle under pneumatic pressure. Current study focuses on this key issue and demonstrated pressure casting process successfully for the fabrication of alumina parts. Slips of a mixture of alumina with different particle sizes in the various proportions and solid loadings were prepared. Slip under PSC resulted in effective interlocking of the particles retaining the shape while de-molding and achieved a sintered density of 98.6 % of theoretical density (TD). Slurry on CSC exhibited a lower sintered density of 97% of TD. Selection of particles with sizes in optimized proportion for PSC results in effective interlocking of particles in green parts as well as grains on sintering as revealed by the microstructure. This leads to higher density and mechanical properties. Slip thus optimized were shaped in to solid spheres of φ 60 mm by PSC targeting grinding applications.

1. Introduction

Slip casting being a wet processing technique offers higher homogeneity of cast parts compared to dry processing techniques. However, poor green density of the cast parts, long duration for cast formation and drying time along with higher rejections limits applications especially for advanced ceramics demanding high productivity [1–3]. Application of pressure while slip casting (PSC) using polymer molds overcome most of these limitations. Thereby, the emerging pressure slip casting technique can be considered as a probable alternative method which offers higher productivity in place of age-old slip casting of ceramics [4–8]. Pressure slip casting proceeds through feeding of the slurry under pre-designed pressure into the polymer mold, holding the cast under pressure for solidification through interlocking of the particles and demolding the cast pneumatically using air pressure at around 4 bars [9–13]. Basic requirement for obtaining densely packed green products is to have a stabilized slip. The influence of deflocculant and the solid loading on the packing behavior of a bimodal particle size distribution indicating the possibility of migration of coarser particles in opposite direction during casting with the consolidated layer acting like sieve [14–16]. Pressure casting is well practiced for traditional ceramic, as most of compositions are based on the clay with plate-like morphology which reinforces through inter-locking to retain the shape while demolding [17–18. Unlike the traditional ceramics, cast formed with advanced ceramics possess irregular morphology and is not favorable for reinforcing itself under pressure and disintegrates while demolding. This limits the wide spread use of pressure casting for technical ceramics and further studies on this subject are also very limited [19–20].

The limited use of alumina in structural applications due to the inherent brittleness had driven the researchers to work towards improving the fracture toughness along with microstructure tailoring to address the issue [21–22]. Hence there is scientific and technological motivation to address these limitations through advanced processing techniques that permit the tailoring of microstructures. In the current study, we have used alumina an advanced ceramic employed for a variety of applications to evaluate the viability of process of pressure casting. Slip using a mixture of alumina with an average particle sizes of 1 and 7 µm in the proportions of 50:50, 40:60, and 30:70 respectively were prepared with a solid loading of 75 wt. % in order to evaluate the effectiveness of interlocking of the particles to retain the shape while de-molding. Slip of alumina in the ratio of 30:70 with solid loading of ≥ 75wt. % on casting under pressure have resulted in the sintered density of 98.6 % of theoretical density (TD). Slurry was also subjected to conventional slip casting for the sake of comparison and exhibited a lower sintered density of 97% of TD.

2. Experimental Procedure

2.1 Characterization of Alumina powders and preparation of the slip:

Two grades (MR-01 & HIM 10) of alumina powder procured from commercial source (M/s. Hindalco, India) was characterized by X-Ray Diffraction (D8-Bruker, Germany) for phase identification and particle size distribution was analyzed by using Laser Diffraction technique (Malvern Instruments Limited, UK). A homogeneously dispersed aqueous slurry of the MR-01 and HM-10 were prepared individual employing Darvan 821A (ammonium polyacrylate; RT Vanderbilt Inc, CT, USA) followed by ultrasonication (VCX 750, Sonics & Materials Inc, CT, USA) was used for particle size measurement. Further the Powder morphologies were also assessed with scanning electron microscopy (SEM) (Hitachi, Tokyo, Japan). In order to carry out casting under pressure and pressure-less conditions an aqueous slip of alumina powders of two average particle sizes were mixed in pre-designed proportions of 50:50, 40:60, 30:70 with solid loading over the range 70–80 wt.%. 1 wt.% of Darvan was used as a dispersant and milled with alumina grinding media for 4 hours on a pot jar mill (20 rpm) to achieve the slips with desired homogeneity and stability. The prepared slips of various solid loadings were studied for their rheological properties using a rheometer (MCR 51, Anton Paar, Austria) at different shear rates by concentric co-axial cylindrical plates setup with 1.75 mm center distance in between them maintaining constant temperature at 25^0C.

2.2 Conventional (CSC) /Pressure Slip Cast (PSC) Processing:

PSC experiments were performed on an industrial model PCM (PCS-100N, SAMA GmbH, Germany) with varying processing control parameters such as feed rate of the slip, slip pressures from 1–40 bar, pressure dwell time and de-molding the cast. Polymer molds for PSC and POP molds CSC were prepared with Stainless Steel (SS) patterns incorporating the shrinkage allowances to obtain the final dimensions of φ 80 mm x 7 mm thickness. These specimens are used for optimizing the sintering parameters and for the preparations of specimens for mechanical characterization as per ASTM standards. Based on the data base generated through these samples, SS patterns were also fabricated to prepare the two-part polymer mold set in order to pressure cast alumina solid spheres of φ 60 mm. Cross sectional views molds used to cast the spherical balls are shown in Fig. 1a) and the polymer molds used for casting Fig. 1b). and the CSC is carried out following the conventional slip casting process using Plaster of Paris (POP) molds. It is evident that unlike CSC, PSC consists of four major operations such as feeding the slip, pressurization, cast formation and cast release.

Several experiments were carried out at our laboratory to achieve the optimum parameters that lead to the cast formation with higher green density values of > 60% of theoretical densities. CSC and PSC samples were sintered at identical conditions at 1600^0C for 1–2 hrs to achieve densities close to theoretical value.

2.3 Characterization of Samples:

Density of the samples (PSC and CSC) was evaluated by Archimedes principle using a density measuring kit (LA 120S, Sartorius AG, Germany) as per ASTM D792, 2013 and hardness values obtained through ASTM C1327. 3-point bend flexural strength was determined using a UTM following ASTM C1161, 2002 and further fracture toughness (K_{Ic}) was determined by ASTM-C1421, (SENB). The samples were also compression tested and compression strength was determined as load per unit area.

3. Results And Discussions

3.1 Characterization of Alumina Powders and Slips:

XRD pattern corresponded to pure alpha Al_2O_3 phase as per alpha phase JCPDS (Match Entry C 96-100-0033) (Fig. 2). Average particle size distribution of alumina powders of grades MR-01, HIM-10 and the powder mix prepared from two grades of alumina are shown in the Fig. 3.

The two grades of the powders have shown an average particle size of 1.43 µm, 7 µm respectively while the average particle size of the powder mix is found to be about 3.16 µm. SEM micrographs showed irregular powder morphology (Fig. 4).

It is evident from the micrograph that the irregular morphology and the size variation as observed in the two grades (finer and coarser) alumina powders are desirable factors in achieving a higher packing fraction and interlocking of the particles one of the critical issues in addressing the collapse of the structure while de-molding the cast part under pressure.

3.2 Rheological behavior:

The parameters required in rheological assessment of the suspensions to achieve the cast bodies with desired slip properties. Among all the slips prepared, only those with ≥ 75 wt. % solid loading (powder mix ratio of 30:70) are observed to be stable and homogenous and studied for the rheology and the plot of Viscosity versus Shear rate as shown in Fig. 5. When a shear force (pressure) is applied on the slip, the particulates have to flow and fill all the cavities in the mold for which a shear thinning behavior is essential. The rheological behavior displayed by the present slip confirms that the lowering of viscosity value which is required for retaining the shape of the cast once pressure is removed. Under the pressure casting conditions, the slip in the polymer-based mold (with average pore size of about 10 μm) has to undergo a forced filtration resulting in the faster thickness built up. This observation is in agreement with finding of the requirement of low viscosity and low yield point for a good control of the casting rate in order to obtain high relative density of the cast bodies.

3.3 Casting of Specimens:

Alumina discs and spheres cast along with pressure casting cycle and sintered are shown in Fig. 6. The green samples immediately after the casting (PSC & CSC) were weighed accurately (± 0.01 g) and kept open in the ambient conditions at an average room temperature of 25^{o}C and A typical pressure cast cycle followed for PSC is shown in the Fig. 6c).

The water content was estimated by the ratio between the weights of the wet body to that of dried one. The results are shown in Fig. 7 where it is evident that the maximum moisture content is 25% and 12% which can be completely removed by open room drying in 24 & 45 hrs. respectively for CSC an PSC samples. The low moisture content in PSC bodies can reduce the time involved in the drying step during the large-scale production with improved productivity and lower rejection rate.

3.4 PSC effect on thickness built up:

All the parameters of the PCS like feed rate, slip pressure, and pressure holding time were monitored carefully for each slip to achieve the maximum green strength. The thickness of the Alumina disc cast samples gradually increases with influence of holding time and under varying applied pressure conditions and is shown in Fig. 8. Thickness built up of the sample is found to be considerably faster till 200 seconds of holding time under given pressure applied on the slip. Then the thickness built up become much slower which is obviously due to the fact that the water finds it difficult to penetrate through as the thick sample layer formed on the internal walls of the polymer mold inhibits the direct path towards the pores. In order to study the maximum thickness built up that can be achieved in the pressure slip casting technique, five different pressures and as long as 300 seconds of (pressure) holding times were studied (Fig. 8).

A flat Alumina block of > 30 mm thick can be pressure slip cast using polymer split molds within 5 min under 35 bar pressure applied on the slip. Accordingly, by fine tuning both the machine and slip parameters it is possible to pressure cast green bodies as large as φ 60 mm (30 mm thick on each part of the split mold) without much difficulty. In the present study, solid Alumina spheres of φ 60 mm were pressure cast reproducibly and sintered to achieve > 98.5% density.

3.5 PSC influence on density and mechanical properties:

The influence of solid loading on the green densities of conventionally cast samples and the same in case of pressure cast samples as a function of the pressure applied on the slip is presented in Fig. 8. This graph highlights the fact that optimum solid loading and pressure to be applied in order to achieve the maximum green density are, 75–80 wt.% and 35 bar respectively. From literature while working on pressure casting of Al_2TiO_5 ceramics observed that green densities over the range 60–65% could be achieved under low pressure on the slip contrary to much higher pressures of > 100 MPa required for the same in CIP (cold isostatic pressing)[13].This can be attributed to the fact that unlike dry pressing, slip with optimum solid loading of 75–80% on pressurization and holding condition selected in our laboratory through several experiments facilitates the re-arrangement of particles in the aqueous medium through rolling, twisting and interlocking finally leading to higher densities. Additionally, the average particle size of 1.43 µm and 7 µm of alumina used in the study is also expected to play a critical role in achieving the high packing factor. It is also evident that solid loading beyond the optimum results in the reduction of green density due to the restriction of movement due to higher particle density for rearrangements of the particles under pressure.

A similar trend is also observed with conventional slip casting due to the gravity settling of the particles rather than cast formation under high solid loading conditions. It is also obvious that due to the removal of excess water under pressure through the porous mold also reduces the drying time and rejection due to warpage and cracks resulting from the differential drying stresses generated during ceramic drying process.

3.5 PSC influence on Microstructural properties:

The microstructural study under FESEM on the sintered alumna samples produced from both the techniques are presented in Fig. 10a) and 10b). The comparison of the both SEM images clearly brings out the close packing and smaller grain size achieved in the pressure cast samples and also supports the higher sintered densities determined in the same. Distribution of smaller grains with an overall average size of 0.514 µm in the inter-particle spaces of coarser-grains leads to better coordination in the pressure cast samples resulting in higher densification in comparison to the conventionally cast samples.

An 22% increase in Vickers hardness is observed with PSC sample (14.92 ± 0.15 GPa) in comparison to CSC samples (11.77 ± 0.15 GPa). This can be attributed to the higher density as indicated by the close packed microstructure with relatively smaller grains. The fracture toughness, flexural strengths of the samples are presented in Table.1 along with densities for the both conventional and pressure cast samples. It is evident that enhancement of the flexural strength from 242.70 to 294.40 MPa, and the fracture toughness from 3.73 to 4.06 $MPa.m^{½}$ in on application for PSC samples which can be attributed to the interlocking of elongated grains (with 3.786 µm major axis, 1.452 µm minor axis) with the smaller grains of average size of 0.514 µm.

4. Conclusions

1. Application of pressure slip casting for technical ceramics with alumina as an example was demonstrated successfully and the critical issue of collapse of cast part while de-molding under pneumatic pressure during PSC cycle was addressed.

2. Slip parameters such as selection of particle sizes, engineering the proportion and optimum solid loading along with instrumental parameters are the key factors for effective interlocking to maintain the structural integrity while de-molding. Pressure casting of Alumina slip with 75wt.% solid loading prepared with mix of powders having d_{50} of 1 & 7 microns in 30:70 ratio has exhibited structural integrity with higher density of 98.5% of TD in comparison to relatively low density of 97% obtained through conventional casting.
3. Enhancement of mechanical properties in pressure cast sample is evident through fracture toughness, flexural strength and Vickers hardness up to 4.06 MPa $m^{1/2}$ and 294.40 MPa and 14.92 ± 0.15 GPa against 3.73 MPa $m^{1/2}$, 242 MPa and 11.77 ± 0.15 GPa respectively in case of conventional cast samples. Enhancement in mechanical properties of PSC samples can be attributed to interlocking of elongated grains (with 3.786 µm major axis, 1.452 µm minor axis) with the smaller grains of average size of 0.514 µm in the inter granular spaces between the elongated grains as is evident from the microstructure
4. Optimized slip and machine parameters have been reconfirmed further by casting φ 60mm alumina solid spheres which upon sintering at 1600^{o}C attained > 98.5% density for the applications as grinding balls. In the current study the pilot scale machine has been utilized to demonstrate the production of 12–14 grinding balls /hr. The productivity can be further increased by introducing multiple cavities in the mold which is presently under investigation.

Declarations

Acknowledgements:

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Tables

Table 1. Density and Mechanical properties of alumina processed by conventional and pressure slip casting:

Process	Density			Mechanical Properties	
	Green Density(g/cc) (% TD)	Sintered Density(g/cc) (% TD)	Flexural Strength (MPa)	Fracture Toughness (MPa $m^{1/2}$)	Hardness (GPa)
Conventional Slip Casting	2.29 (57%)	3.873 (97%)	242.70	3.73	11.77±0.15
Pressure Slip Casting	2.55 (65%)	3.931(98.6%)	294.40	4.06	14.92±0.15

Figures

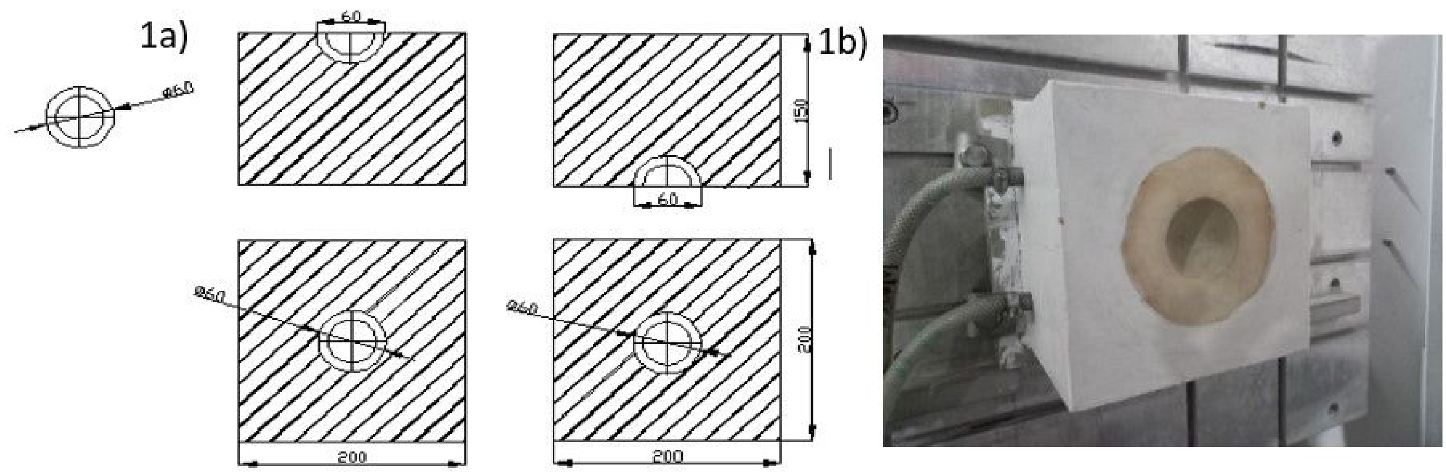

Figure 1

a) Cross sectional views molds and the polymer molds fabricated for casting of spherical grinding balls and b) Polymer mold for casting solid sphere of Ø 60 mm

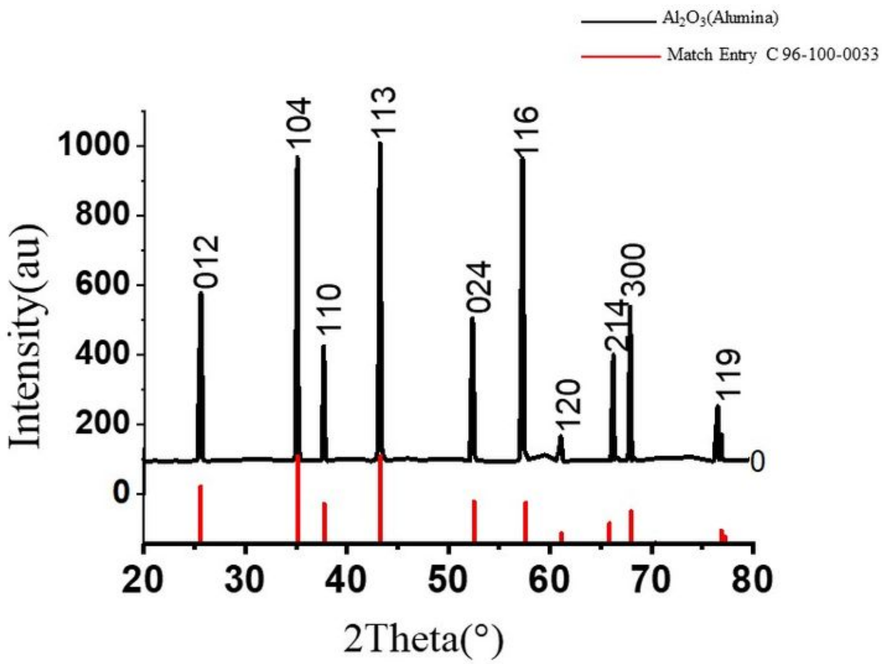

Figure 2

XRD pattern corresponded to pure alpha Al2O3 phase

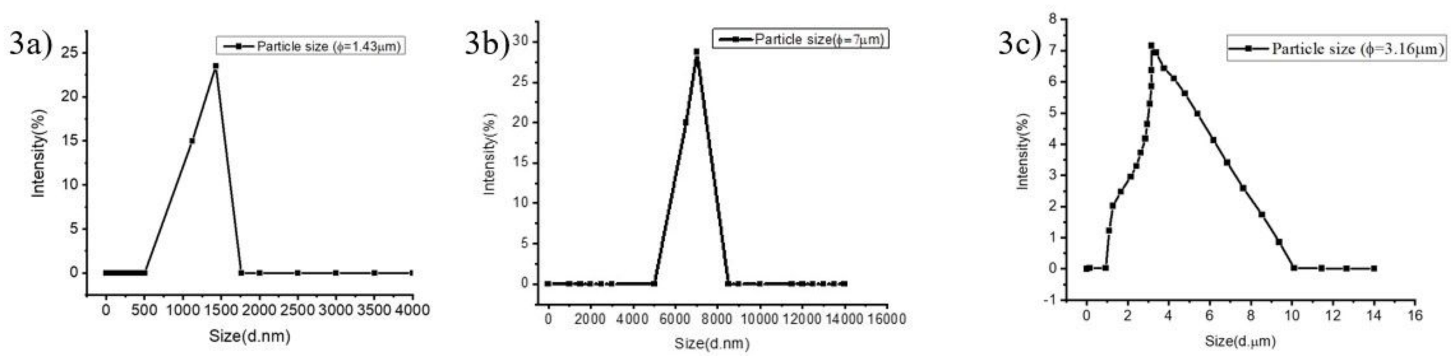

Figure 3

Graphs showing particle size distribution of a) MR-01, b) HIM-10 and c) mix of both the powder

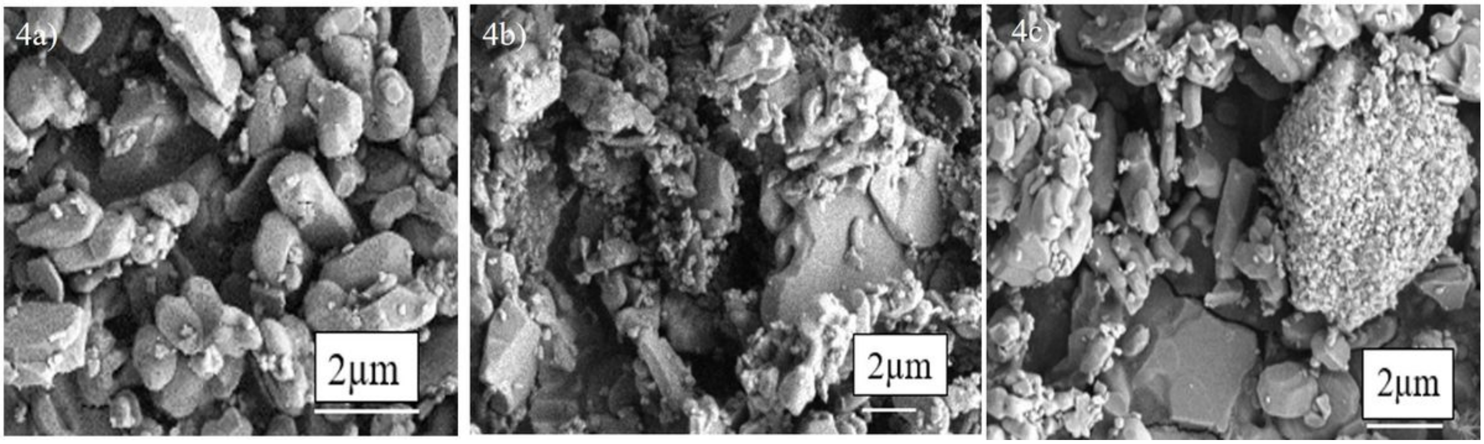

Figure 4

Morphology as observed through FESEM for a) MR 01, b) HIG 10 and c) mix of both a) & b)

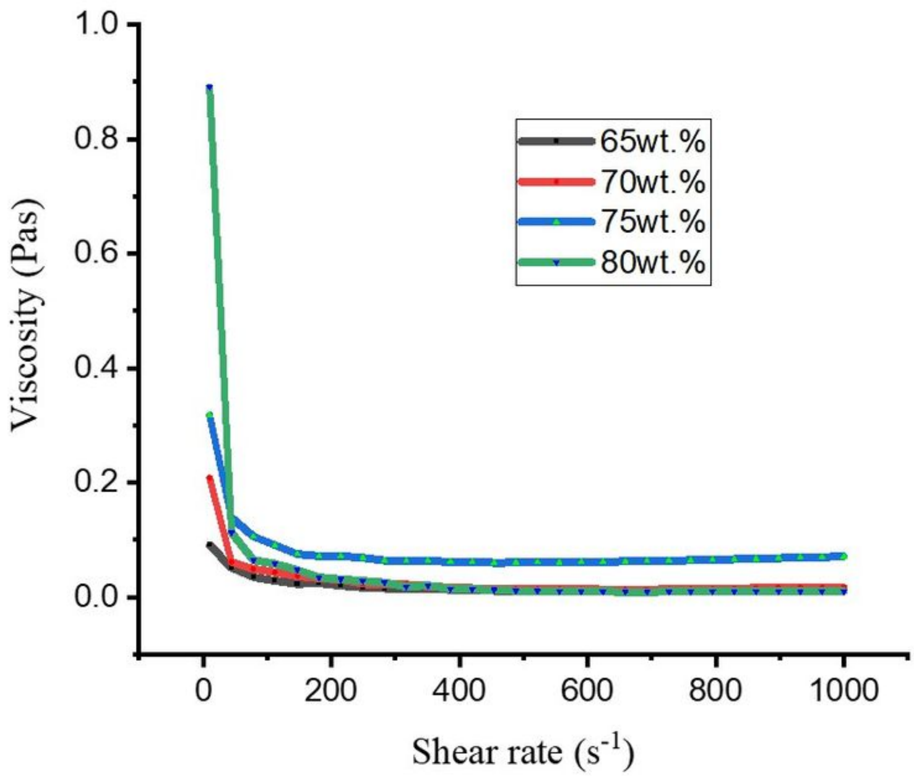

Figure 5

Rheological behavior of Alumina slips at various solid loading conditions.

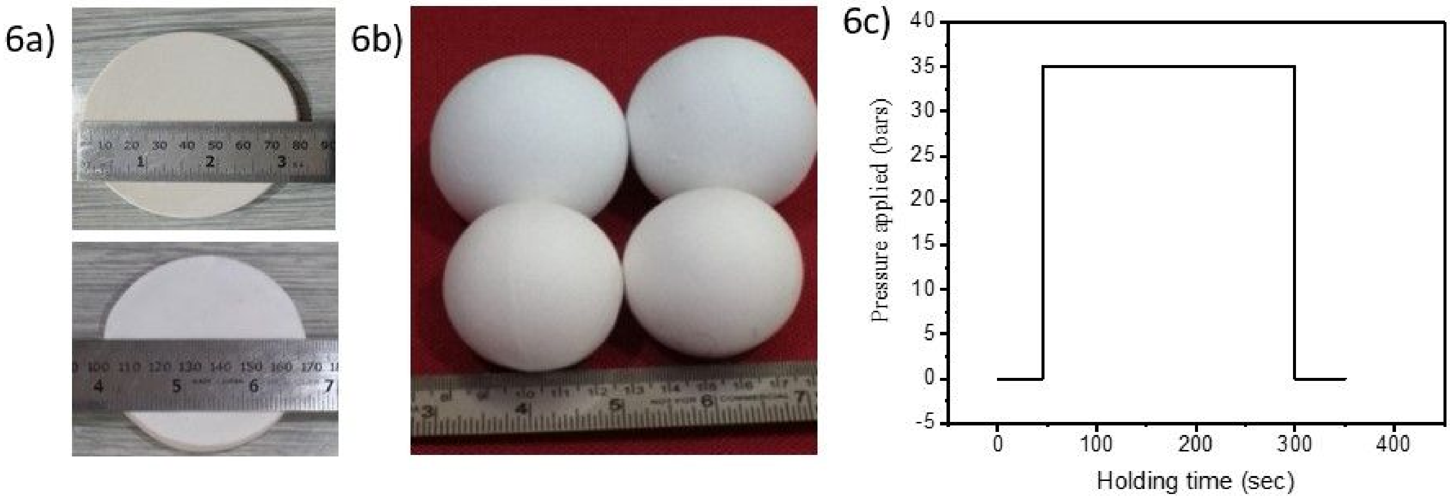

Figure 6

The pressure cast green and sintered samples of a) disc & b) solid sphere and c) a typical pressure cast cycle followed in the present work

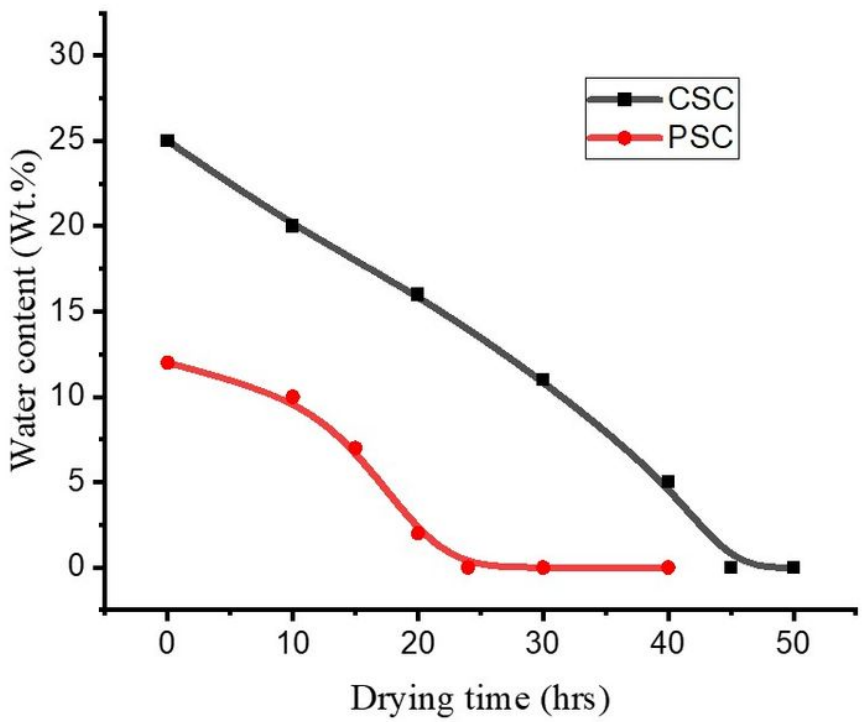

Figure 7

PSC and CSC Alumina Cast Samples Drying behavior.

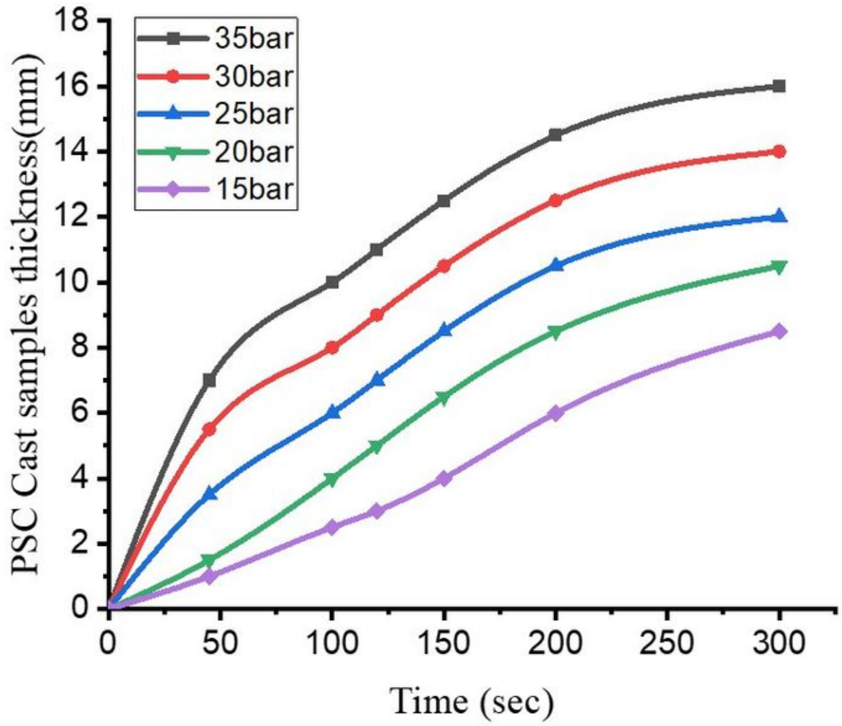

Figure 8

Alumina Cast sample thickness built up under applied pressure conditions as a function of holding time.

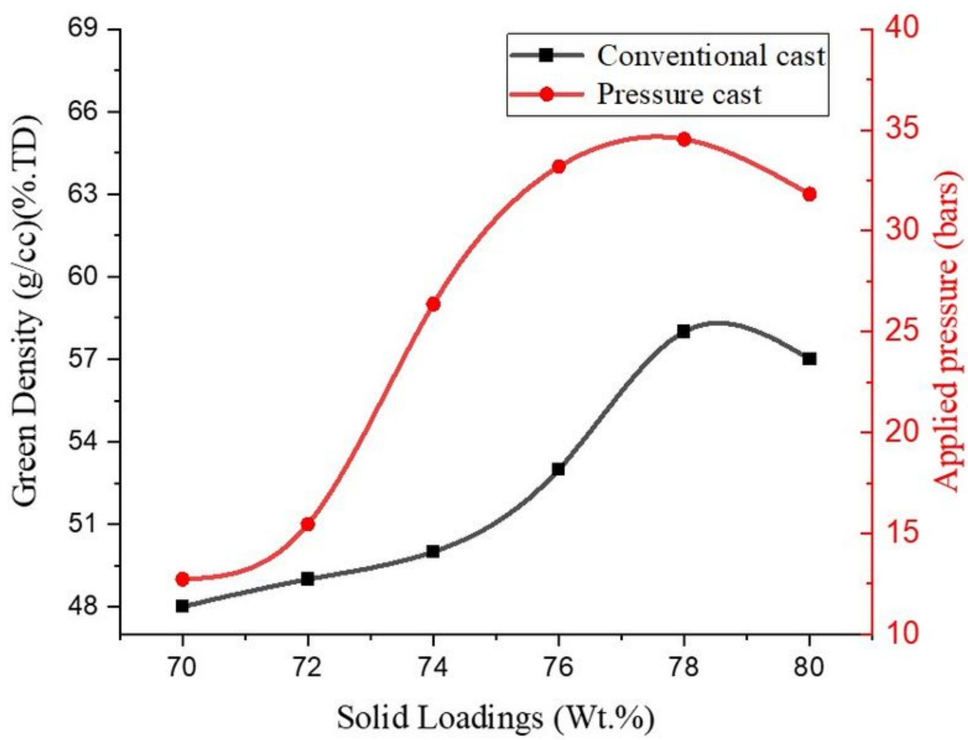

Figure 9

Green density Variation through conventional and pressure casting processes.

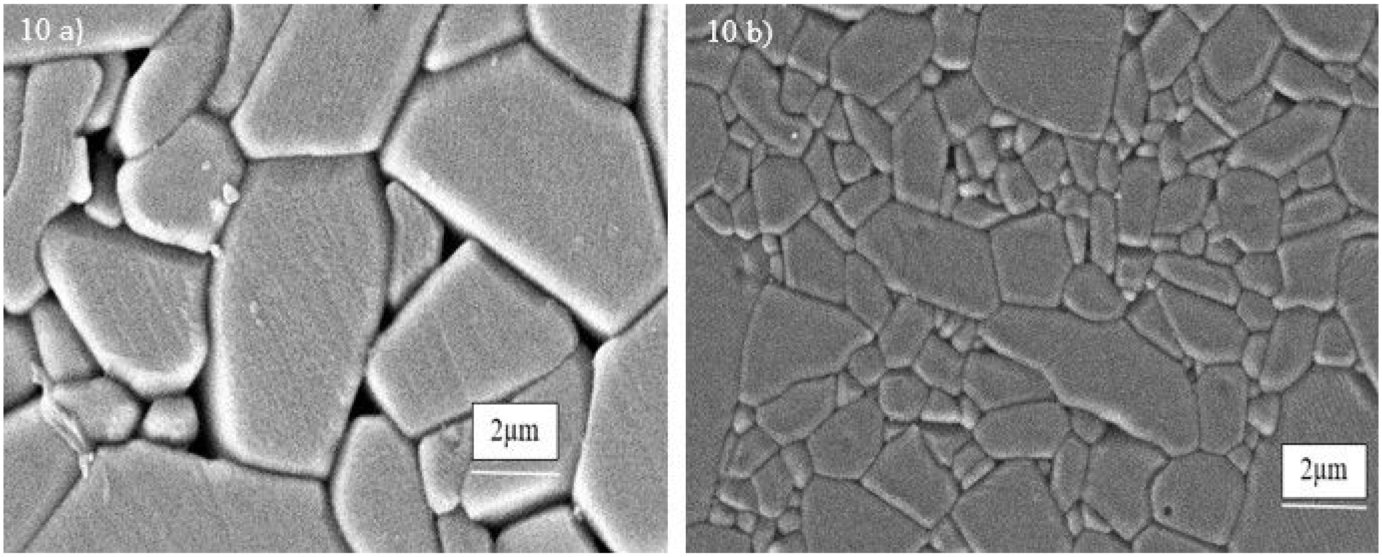

Figure 10

FESEM scan (under same magnification) of sintered Al_2O_3 samples (30:70) fabricated by a) CSC and b) PSC