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Pressure slip casting and cold isostatic pressing of aluminum titanate green ceramics: A comparative evaluation

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Abstract

Aluminum titanate (Al_2TiO_5) green bodies were prepared from mixture of titania and alumina powders with different particle sizes by conventional slip casting (CSC), pressure slip casting (PSC) and cold isostatic pressing (CIP). Precursor-powder mixtures were evaluated with respect to the powder properties, flow behaviours and shaping parameters. Green densities were measured and correlated with the fractographs. A substantial increase in green densities up to 60 %TD (theoretical density of 4.02 g/cm^3, calculated based on rule of mixtures) is observed with the application of 2–3 MPa pressure with PSC. While particle size distribution and solid loading are the most influential parameters in the case of CSC, with PSC pressure also plays a key role in achieving the higher green densities. Being a dry process, high pressure of > 100 MPa for CIP is essential to achieve densities in the range of 60–65 %TD. Slip pressurization under PSC conditions facilitate the re-arrangement of particles through rolling, twisting and interlocking unlike CIP processing where pressure is needed to overcome the inter-particle friction.

Keywords: *aluminium titanate ceramics, shaping, slip casting, pressing, thermal applications*

I. Introduction

Defect free near net shaping of advanced ceramic powders into engineering components in large scale manufacturing has always been a challenge to ceramic engineers [1]. Among the powder compaction processes, isostatic pressing under cold (CIP) or hot (HIP) conditions is expected to provide parts with high density as well as isotropic properties [2,3]. Colloidal processing techniques such as injection moulding and slip casting are two existing large fabrication processes generally practiced; however the application is limited by several factors [1–4]. Injection moulding essentially requires large amount of binders or polymers and pyrolysis of the organic polymers becomes cumbersome especially for larger components [2,5]. Slip casting proceeds by capillary forces which correspond to 0.1 to 0.2 MPa leading to longer formation time and drying durations. Plasters of paris mold contamination in the cast and solid waste generation due to frequent mold rejection are other issues [6–8]. Main issue of low productivity of slip casting can be addressed to the application of external pressure. Fluid flow through the porous medium with interconnected pores under pressure obeys Darcy’s differential equation:

$$S^2 = (2\Delta P/ \eta\cdot\rho_p\cdot R_s) \times (\O_c/(\O_c–\O_s))\cdot t$$

where, S is the cake thickness, ΔP - pressure of slurry, η - viscosity of the slurry, ρ_p - particle density, R_s - permeability of the cake, $\O_c$ - solid volume fraction of the cake, $\O_s$ - solid volume fraction in the slurry and t - the casting time. Practically, casting rate is controlled by the resistance of cast layer and the mold thickness. Further, the cake is expected to grow at a faster rate to a peak value followed by a decrease exhibiting a parabolic behaviour with respect to other variables [9–12].

However, pressure casting in durable polymer moulds with average pore sizes of a 10 µm does not de-

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pend on capillary forces but on external pressure applied to slurry isostatically up to 6 MPa, which substantially enhances the isotropy of properties and productivity. This innovative tool also permits the mechanization of the process providing high efficiency. Pressure casting is nowadays well established in the traditional ceramic processing with respect to hollow and solid shapes. The pressure casted parts are demonstrated to have the additional advantages of reduced water content of the cast, better handling and green strength [13–15].

In the area of advanced ceramics attempts are now still in progress. However, achieving suitable rheology of the slurry for pumping, filtration behaviour under pressure and demoulding of the cast articles are critical issues. Unlike traditional clay based ceramics, advanced ceramics leads to the improper alignment of particles, weak mechanical interlocking and collapse of the cast during demoulding [16].

The objective of the current work is, therefore, to comparatively evaluate conventional slip casting (CSC), pressure slip casting (PSC) and cold isostatic dry pressing (CIP) of a precursor-powder mixtures used for preparation of Al_2TiO_5 ceramics. The precursor-powder mixtures consist of a stoichiometric amount of alumina (Al_2O_3) with different particle sizes and titania (TiO_2), as well as 5 wt.% of talc as additive for suppressing the eutectoid decomposition of Al_2TiO_5 [17–19]. Aluminum titanate, due to its inherent low thermal expansion coefficient, high thermal shock resistance and low thermal conductivity finds potential applications as diesel particulate filters [20,21], riser tubes and nozzles for handling liquid aluminium melts and as non-wetting surfaces for molten non-ferrous melts [22]. It has a low bulk thermal expansion and is used in automotive engine exhaust pipe, thermocouple protecting sheath and as crucible [23–25]. Properties exhibited by the cast parts under pressure (PSC) and pressureless conditions (CSC) are compared with isostatically dry formed CIP specimens and correlated to the processing parameters and forming techniques.

II. Experimental procedure

2.1 Characterization of raw materials and mixtures

Alumina powders obtained from two different sources (CRF-30, Baikowski, France and MR-01 and MR-06, NALCO, India), titania (TiO_2, Qualigens, India) and magnesium silicate ($Mg_3Si_4O_{10}(OH)_2$) as stabilizing additive were used in the study. The powders were characterized by X-ray diffraction studies for phase analysis (D8 advanced, Bruker, Germany) and particle size and morphology by scanning electron microscope (SEM, S-4300SE/N, Hitachi, Japan). The precursor-powder mixtures in the Al_2TiO_5 stoichiometry were prepared using alumina (Al_2O_3) and titania (TiO_2) molar ratio of 1 : 1 and with the optimized concentration of 5 wt.% of $Mg_3Si_4O_{10}(OH)_2$, as reported in our earlier studies [17–19]. The samples are designated as shown in Table 1.

2.2 Preparation and characterization of slurries

The prepared powder mixtures were dispersed in distilled water using 1 wt.% Darvan 821A (R.T. Vanderbilt Co., Inc., USA) as dispersant. The suspensions were then milled for 2 hours in a rubber lined ball mill using alumina balls at 1 : 1 charge to balls ratio to achieve optimum solid loading of precursor mixtures in the range of 63–65 wt.% depending upon the formulation. Rheological behaviour of the slurries was measured at varying shear rates using a Rheometer (MCR 51, Anton Paar rheometer).

2.3 Casting of the slurry

The casting was carried out using a pressure casting machine model PCM-100 (SAMA) with a closing force of 1000 N and a maximum operating pressure up to 4 MPa. The mold was fabricated with polystyrene beads and additives as per the proprietary techniques of M/s. SAMA Maschinenbau GmbH. A prefabricated SS-316 of pattern of 100 mm diameter × 8 mm thickness with shrinkage allowance of 20 % for the cast part and proper release angle was used to produce polymer (SAMA-pore) macro porous moulds. The moulds were characterized by SEM and micrographs were recorded. The ALT-1, ALT-2 and ALT-3 slurries were pumped into the mould (as fabricated above) with a feed pressure of 5 bar and a filling time of 15 seconds. After filling, a slip pressure of 1.0–3.5 MPa was applied with an increment of 0.5 MPa and was maintained for 60 seconds to determine the effect of the pressure on build up of the cast. After opening of the mould the cast was removed instantly by vacuum holder. For the sake of comparison the same batches ALT-1, ALT-2 and ALT-3 slurries were also cast through conventional slip casting generally practiced in ceramic processing.

2.4 Granulation, flow behaviour and cold isostatic pressing

The precursor-powder mixtures ALT-1, ALT-2 and ALT-3 were mixed with polyvinyl alcohol (PVA) as a binder (2 wt.%). The obtained powders were granulated and powder flow measurement was carried out using

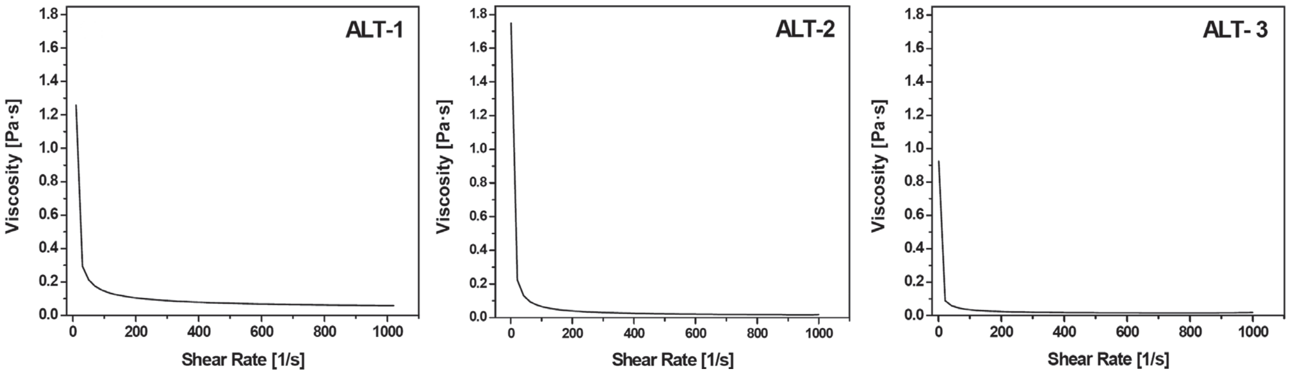
Table 1. Designations of precursor-powder mixtures

Sample notation	Alumina (Al_2O_3)	Titania (TiO_2)
ALT-1	CRF-30	TiO_2
ALT-2	MR-01	TiO_2
ALT-3	MR-01 + MR-06	TiO_2

Table 2. Specification of raw materials

Sample notation	Raw materials	Average particle size (D_{50}) [μm]	Phase composition	Morphology
ALT-1*	CRF-30	0.3	>85 % α- +15% γ-alumina	Irregular
	TiO_2	0.3	100% anatase	Irregular
ALT-2*	MR-01	1.0	100% α-alumina	Irregular
	TiO_2	0.3	100% anatase	Irregular
ALT-3*	MR-01	1.0	100% α-alumina	Irregular
	MR-06	6.0	100% α-alumina	Irregular
	TiO_2	0.3	100% anatase	Irregular

*5 wt.% of magnesium silicate additive was added in all samples



a) b) c)

Figure. 1 Viscosity vs. shear rates of: a) ALT-1, b) ALT-2 and c) ALT-3 slurries

an analyser attached to a Texture Analyser (Stable Micro System, UK), which utilizes a rotating blade moving down and up through the granules achieving the controlled flow. The procedure is described elsewhere [26,27]. Granules with acceptable flow behaviour were compacted by uniaxial pressing with a pressure of 55 MPa. The sample ALT-1 was vacuum sealed in plastic bags and isostatically pressed at 50, 100 and 140 MPa using a cold isostatic press (Avure Technologies, USA), with the pressure of 100 MPa selected as the optimum. Further, the ALT-2 and ALT-3 samples were compacted at 100 MPa.

2.5 Drying and characterisation of samples

All specimens were dried in an oven at 110 °C and subjected to green density determination by dimensional method (mass/volume). The microstructural features of green samples were observed by SEM (S-4300SE/N, Hitachi, Japan).

III. Results and discussion

Average particle size (D_{50}), phase composition from XRD analysis and morphology of the raw materials are given in Table 2. CRF-30 alumina confirms the presence of α- and γ-alumina phases with α-Al_2O_3 as the major phase with an average particle size of 0.3 μm and irregular morphology. MR-01 and MR-06 alumina have shown 100% α-Al_2O_3 with average particle sizes of 1.0 and 6.0 μm, respectively, with an irregular morphology. Titania exhibited single phase of anatase with average particle size of 0.3 μm. Magnesium silicate was used as a common additive in all powder mixtures.

3.1 Solid loadings and rheological behaviour of slurries

Optimum solid loading and viscosity at constant shear rate for the slurries ALT-1, ALT-2 and ALT-3 are shown in Table 3. Figure 1 shows the change in viscosity with varying shear rates of the slurries ALT-1, ALT-2 and ALT-3, respectively. It is evident that all the slurries have a pseudoplastic behaviour at lower shear rates and as the shear rate is increased beyond 100 s^{-1}, the slurries exhibit almost Newtonian behaviour adaptable for casting of the slurry.

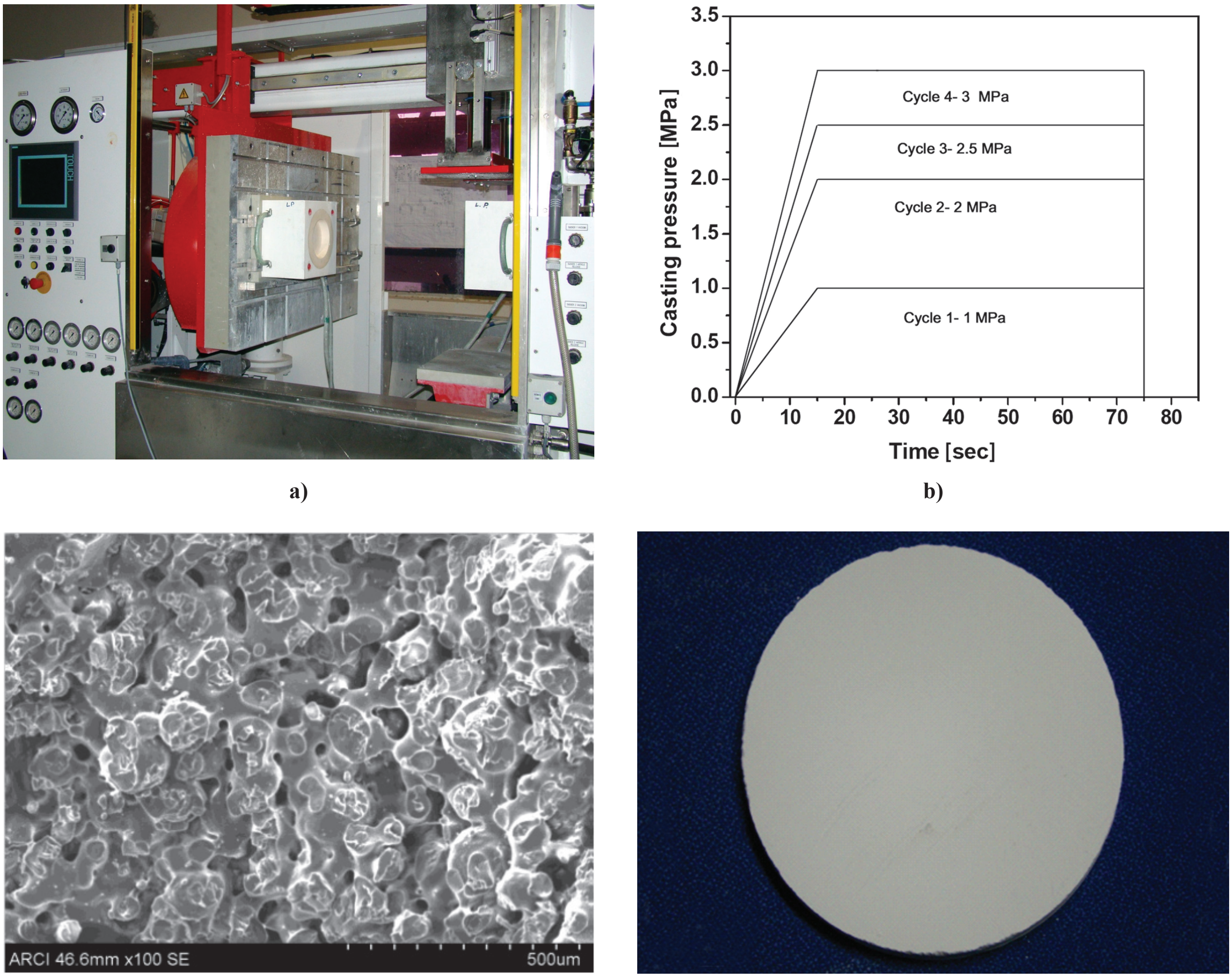
A low viscosity of the slurry results in a slow casting rate and cracks. However, high viscosity of the slurry results in pin holes and poorly drained surfaces generally in the case of CSC. In case of PSC the mechanism being pressure filtration, the suspensions are subjected to isostatic compression shear although shear rheology is still important.

3.2 Pressure casting of slurry

Pressure cast machine fitted with the mould is shown in Fig. 2a and the casting cycles are shown in Fig. 2b. A typical cast part is also shown in Fig. 2d. The pressure cast polymer moulds are prepared from SAMA-pore technologies and SEM micrograph of the mould is shown in Fig. 2c. SEM micrographs clearly exhibit the interconnected pores which are responsible for the permeability

Table 3. Solid loading and viscosity

Sample notation	Solid loading [%]	Viscosity [Pa/s]
ALT-1	63	1.3
ALT-2	65	1.7
ALT-3	65	0.93



a) b)

c) d)

Figure 2. Pressure casting machine fitted with polymer mould (a), pressure casting cycles (b), SEM micrograph of polymer used for preparation of mould (c) and cast part (d)

of water under pressure. The initial cake formation along with the interconnected pores results in effective particle separation from the slurry leading to the cast formation. PSC is carried out using a PLC based casting cycle as per the programme pre-set parameters. When the pressure is applied on the slurry, the volume fraction increases the inter-particle repulsion and the osmotic pressure of the suspension increases. However, the optimum amount of surfactant layer allows a gradual increase in volume fraction under compressive stress field. It is well known that compressive properties of colloidally stable suspensions are reversible. Nevertheless, as the isostatic stress is above the compressive yield stress, the particle network forms resulting in consolidation.

3.3 Cold isostatic pressing

The cohesion index is regarded as the ratio of cohesion coefficient to the weight of the sample evaluated and is calculated by integrating the negative areas under the force vs. displacement curves obtained through flow behaviour estimation. A cohesion index of 11–14 for all the samples ALT-1, ALT-2 and ALT-3 corresponds to free flowing behaviour (categorisation is described elsewhere) and is suitable for compaction [26,27]. Pressure cycles (Fig. 3a) employed along with a typical compacted sample of ALT-1 is shown in Fig. 3b.

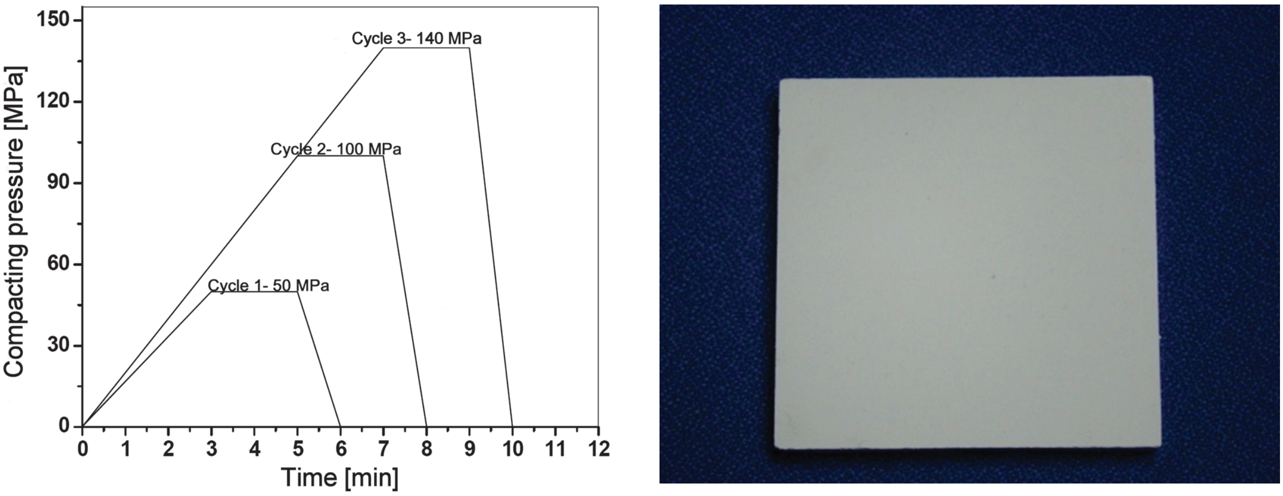
3.4 Characterisation of green specimens

Pressure applied to achieve the maximum green density, in case of ALT-1, ALT-2 and ALT-3 samples

Table 4. Comparison of PSC and CIP specimens

Sample notation	Pressure slip casting (PSC)		Cold Isostatic Pressing (CIP)		Increase in green density CIP vs. PSC [%]
	Pressure p_{max} [MPa]	Green density ρ_{GD} [%TD]	Pressure p_{max} [MPa]	Green density ρ_{GD} [%TD]	
ALT-1	3	44.0	100	58.5	33.0
ALT-2	3	51.8	100	58.7	13.2
ALT-3	2	60.1	100	65.0	8.2

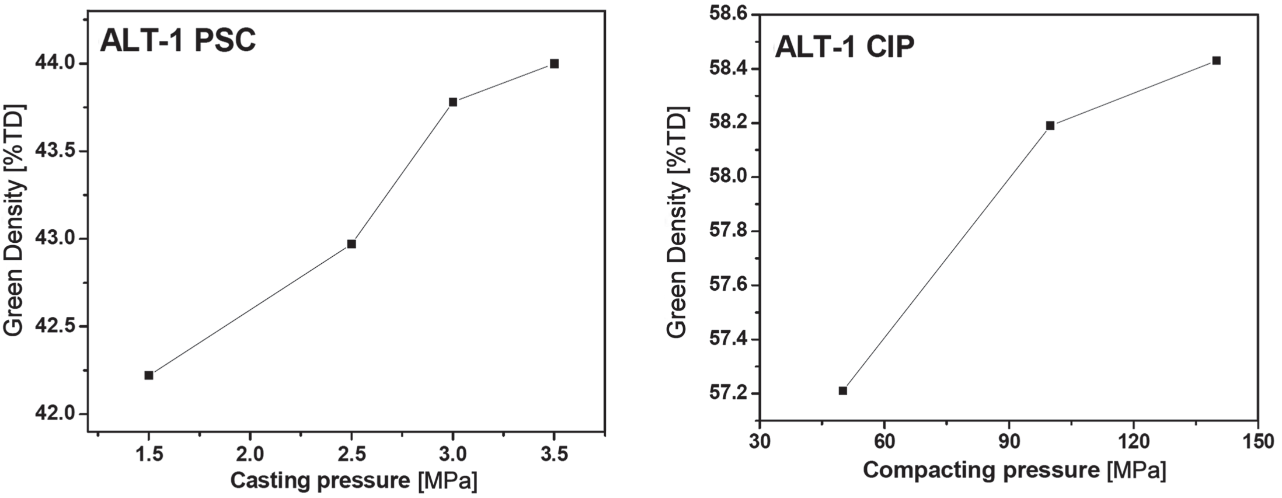
p_{max} - pressure used to achieve maximum green density, ρ_{GD} - maximum green density achieved,



a) b)

Figure 3. Pressure cycles in CIP (a) and compact part ALT-1 (b)

and the maximum green density achieved for these samples with respect to pressure slip casting (PSC) and cold isostatic pressing (CIP) are shown in Table 4. Further, a comparative evaluation of increase in the green density is depicted in Table 4. Plots of pressure versus green density (GD) in case of ALT-1 samples for PSC and CIP conditions are shown in Fig. 4. It is evident that CIPing of the uni-axially pressed ALT-1 samples at a pressure of 50 MPa has shown an increase in percentage of green density by 30 % (theoretical density of 4.02 g/cm^3 is calculated based on rule of mixtures of alumina - 3.986 g/cm^3 and titania - 4.25 g/cm^3) in comparison to PSC. On increasing the pressure to 100 MPa the increase in the green density is around 33 % and further pressure increase to 140 MPa has shown only negligible effect in percentage increase in the green density. Increase in percentage green density for ALT-2 and ALT-3 samples on CIPing at 100 MPa (optimum pressure based on CIPing of ALT-1 sample) is found to be less significant with re-



a) b)

Figure 4. Plots of green density vs. pressure for ALT-1 under: a) PSC and b) CIP condition

Table 5. Comparison of PSC and CSC specimens

Sample notation	Pressure slip casting (PSC)		Conventional slip casting (CSC)	Increase in green density CIP vs. PSC [%]
	Pressure p_{max} [MPa]	Green density ρ_{GD} [%TD]	Green density ρ_{GD} at atmospheric pressure [%TD]	
ALT-1	3	44.0	43.0	-
ALT-2	3	51.8	48.3	7.3
ALT-3	2	60.1	50.3	19.4

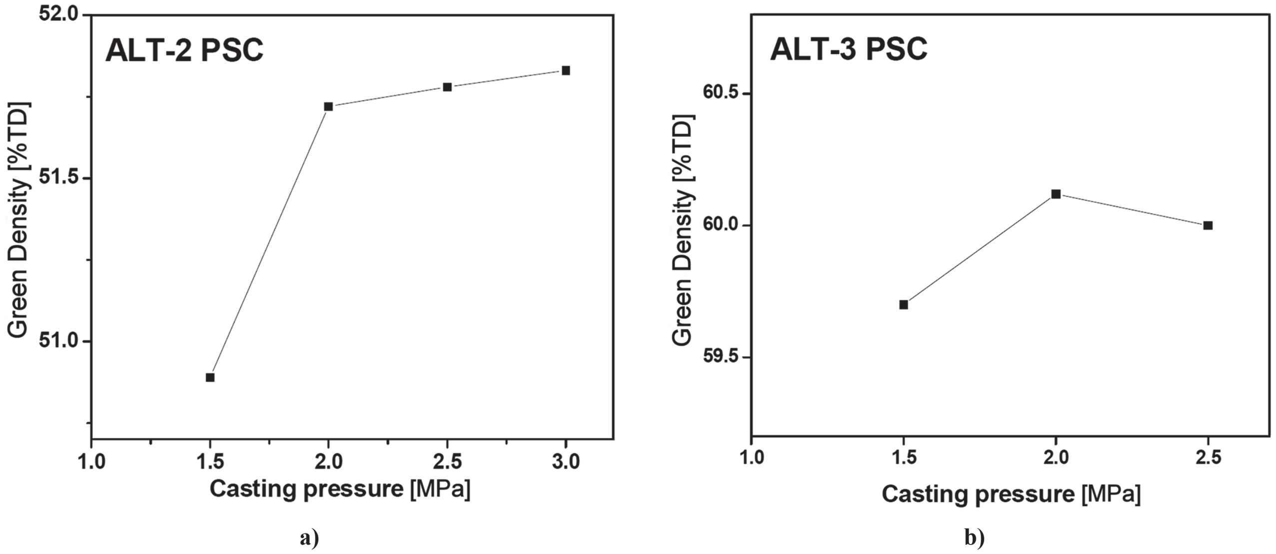
p_{max} - pressure used to achieve maximum green density, ρ_{GD} - maximum green density achieved,

spect to ALT-1 samples. The samples ALT-2 and ALT-3 exhibited only 13.2 % and 8 % increase in density in comparison to the PSC samples. As the applied pressure (100 MPa) is well below the yield strength of the alumina particles, applied pressure goes to overcome the inter-particle friction and porosity decreases as the pressure exceeds 50 MPa by the rearrangement of the particles through rolling, twisting and packing under pressure. Rearrangement is more effective for the sample ALT-3 formulated from a mixture of alumina with particle sizes of 0.3, 1 and 6 µm.

Table 5 shows a comparison of green densities of slip casting specimens with pressure slip casting. It is evident that in case of ALT-1 sample, no significant change in green density is observed by processing with CSC or PSC method (even at a pressure of 3 MPa for PSC process). On the contrary, ALT-2 and ALT-3 samples have exhibited 7.3 % and 19.4 % increase in green density on application of 3 and 2 MPa pressure with PSC processing in comparison to CSC.

Plots of GD versus pressure for ALT-1 are already presented in Fig. 4a and for the samples ALT-2 and ALT-3 under PSC conditions are shown in Fig. 5. The sample ALT-1 achieved a maximum green density of 44 %TD at 3 MPa pressure; however, the samples ALT-2 and ALT-3 have shown a maximum density of 51.8 and 60.1 %TD at a lower pressure of 2 MPa. On further increasing the pressure beyond 3 MPa in the case of ALT-1 and 2 MPa in the case of ALT-2 results have shown negligible effect on green densities (exhibiting a plateau). Unlike ALT-1 and ALT-2, the sample ALT-3 has clearly indicated a decrease in green density with further increase in pressure. Colloidal processing such as slip casting involves particles in the slurry state and the interparticle frictions are negligible in case of optimum dispersions of the particles. However, van der Waals forces become prominent. It is evident that the effect of pressure on the sample ALT-1, with 0.3 µm particle sizes is minimal resulting in the same density as in the CSC samples even at 3–3.5 MPa. The effect of pressure becomes evident with the PSC- ALT-2 and ALT-3 samples exhibiting an increase of green density by 7.3 and 19.4 % in comparison to the CSC samples. The results also correlate well with the particle size and distribution. A decrease in green density for ALT-3 beyond the applied pressure of 2 MPa can be attributed to the delamination of cast layers formed under pressure after achieving relatively high green density of 60 %TD.

Microstructure of the fracture surfaces were examined under scanning electron microscope and the representative microstructures of the samples ALT-1, ALT-2 and ALT-3 processed through CIP, PSC and CSC with highest green densities are shown in Fig. 6. There



a) b)

Figure 5. Plots of green density vs. pressure for: a) ALT-2 and b) ALT-3 samples under PSC condition

is clear distinction between the surfaces depending on the forming process. All CIP samples have shown (Fig. 6a,b,c) high degree of roughness with significant particle agglomeration and lesser pores. The samples ALT-3 have shown less agglomeration and absence of larger flaws and more homogeneity complementing high green density (65 %TD) observed for the sample.

A more homogeneous and uniform distribution of particles in comparison to CIP, is evident for PSC samples. The samples PSC and CSC exhibited more of a layered structure and seem like glued together with interlayer voids. A more prominent effect of pressure is evident for PSC ALT-3 (Fig. 6f) in comparison to CSC ALT-3 samples (Fig. 6i), which correlates well with high green density.

IV. Conclusions

Titania and alumina precursor oxide mixtures (in the aluminum titanate stoichiometry), with different particle sizes were subjected to slip casting under pressureless (CSC) and pressure (PSC) conditions including cold isostatic pressing (CIP). Conventional slip casting (CSC) using slurries having similar solid loadings and pseudoplatic behaviour and green densities of 43, 48 and 50 %TD for ALT-1, ALT-2 and ALT-3 samples, respectively, revealed that the particle size distribution of alumina play a key role in achieving the high green densities. Pressure slip casting (PSC) with the same batch of CSC slurries enhanced the green densities to 44, 51 and 60 %TD for ALT-1, ALT-2 and ALT-3 samples. Applity of pressures of 2 to 3 MPa under PSC conditions reveals the combined effect of particle size distribution and applied pressure on green densities. However, ALT-1 has shown only minimal effect on green densities on application of pressure in comparison to CSC, due to the repulsive interparticle van der Waals forces associated with 0.3 μm alumina in the colloidal state. PSC involves the slip pressurization which in turn facilitate the rearrangement of particles through rolling, twisting and interlocking leading to the cast bodies at substantially lower pressures in comparison to CIPing. Requirement for high applied pressure of 100 MPa and above while CIPing can be attributed to the need to overcome the inter-particle friction within the dry precursor mixes and simultaneous minimization of porosity in the green bodies. Higher densities of 60 %TD through PSC and a maximum of 65 %TD through CIP for ALT-3 samples are attributed to the proper blending of particles with

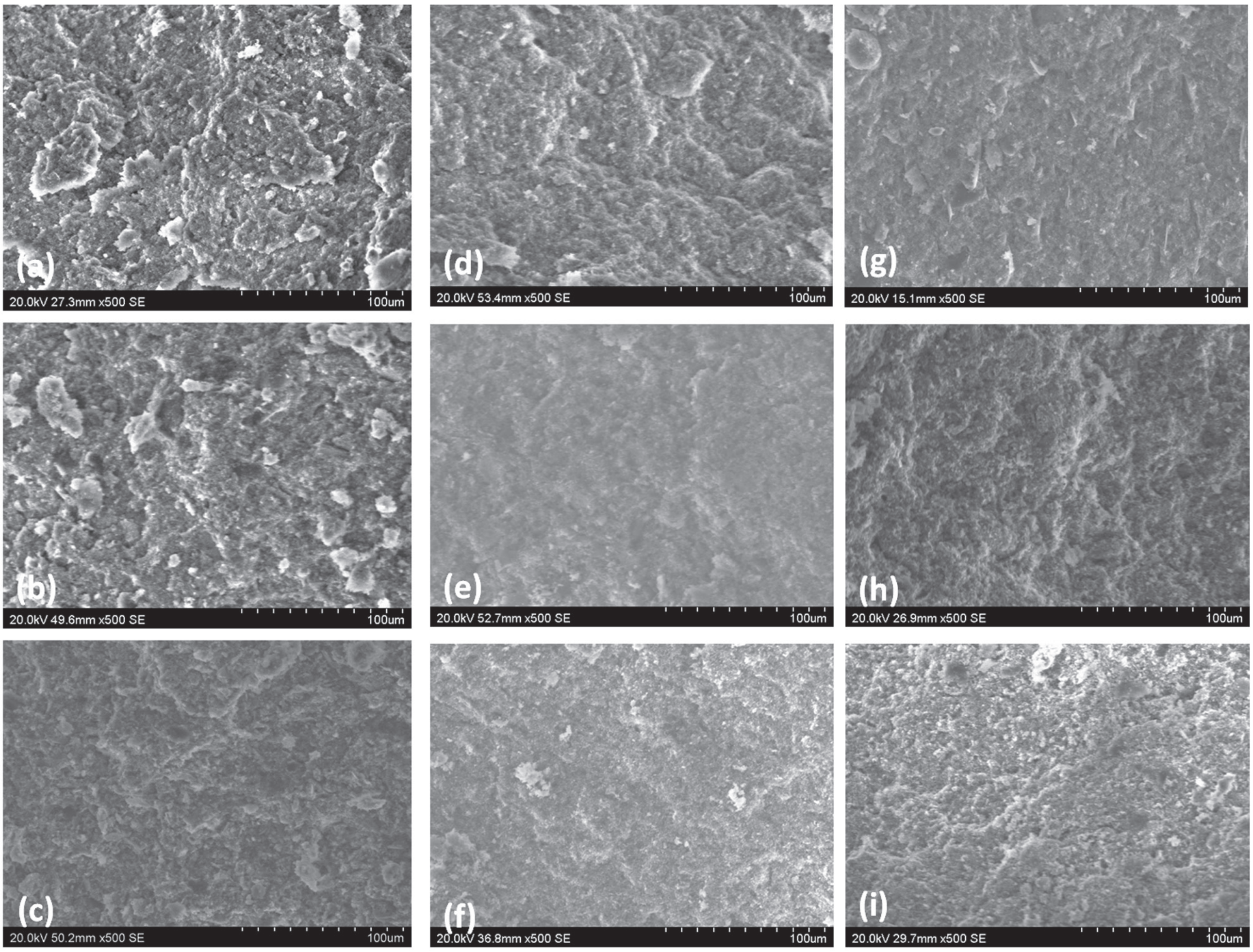


Figure 6. SEM micrographs of: CIP (a) ALT-1, (b) ALT-2 and (c) ALT-3; PSC (d) ALT-1, (e) ALT-2 and (f) ALT-3; and CSC (g) ALT-1, (h) ALT-2 and (i) ALT-3 samples

a variety of sizes (0.3, 1 and 6 µm), achieving a close packing under applied pressure.

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