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Centrifugal Slip Casting of Zirconia (TZP)

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Abstract

Tetragonal zirconia (3Y-TZP) ceramic parts were produced via centrifugal slip casting. This forming method combines the advantages of wet powder preparation with a very efficient densification technology to produce massive and near-net-shape ceramic parts. Aqueous TZP slurries with solids contents up to 42·3 vol.% and low viscosity were prepared using Darvan C as a deflocculant and ultrasonic cavitation to achieve a good homogenization. At solids loadings above 40 vol.% differential segregation effects during centrifugation are negligible. Very good particle packing with narrow pore size distributions (from $r_{25} = 21\cdot 1$ *nm to* $r_{75} = 17\cdot 3$ *mm) were achieved. High green densities of up to 57% of theoretical density (TD) were obtained after centrifuging at 4000 g for 90 min. High sintered densities (>99·3% TD) were achieved at sintering temperatures as low as 1300°C with 2 h holding time. Sintered compacts have a very fine microstructure with an average grain size of 0·3 μm and narrow grain size distribution. Centrifugal slip casting has the potential to produce massive ceramic parts with reduced defect sizes and thus high reliability.*

Keramische Bauteile aus tetragonalem Zirkonoxid (3Y-TZP) wurden über Zentrifugalschlickerguss hergestellt. Diese Formgebungstechnologie verknüpft die Vorteile der kolloidchemischen Pulveraufbereitung mit einer effizienten Verdichtungsmethode zur Herstellung von massiven und endformnahen keramischen Teilen. Wässrige TZP-Suspensionen mit bis zu 42·3 Vol.% Feststoffgehalt und niedriger Viskosität wurden durch Zugabe von Darvan C als Verflüssiger aufbereitet und mittels Ultraschallbehandlung homogenisiert. Bei Feststoffgehalten über 40 Vol.% ist eine differentielle Sedimentation während des Zentrifugiervorganges vernachlässigbar. Sehr gute Teilchenpackungen mit engen Porengrössenverteilungen (von $r_{25} = 21\cdot 1$ *nm bis* $r_{75} = 17\cdot 3$ *nm) wurden erhalten. Nach einer Zentrifugierzeit von 90 min bei 4000 g wiesen die Grünkörper Dichten von bis zu 57% der theoretischen Dichte (TD) auf. Hohe Sinterdichten (>99·3% TD) wurden schon bei Sintertemperaturen von 1300°C mit einer Haltezeit von 2 h erreicht. Gesinterte Teile haben ein feines Gefüge mit einer mittleren Korngrösse von 0·3 μm und einer engen Korngrössenverteilung. Der Zentrifugalschlickerguss hat das Potential zur Herstellung massiver Keramikbauteile mit reduzierten Defektgrössen und damit hoher Zuverlässigkeit.*

Des céramiques de type 3Y-TZP sont préparées par coulage centrifuge. Cette technologie de mise en forme combine les avantages de la chimie des colloïdes pour la préparation des poudres avec une méthode efficiente de densification afin d'obtenir des céramiques massives ayant des dimensions proches de celles des pièces finales. Des suspensions aqueuses de TZP contenant jusqu'à 42·3 vol.% de phase solide et une faible viscosité sont obtenus par addition d'un défloculant (Darvan C) et sont homogénéisées à l'aide d'ultrasons. Lorsque le contenu en phase solide dépasse 40 vol.%, la sédimentation différentielle est négligeable. On obtient un entassement compact des particules avec une distribution étroite de la taille des pores (de $r_{25} = 21\cdot 1$ *nm à* $r_{75} = 17\cdot 3$ *nm). Après 90 min de traitement centrifuge à 4000 g, les pièces atteignent une densité verte allant jusqu'à 57% de la valeur théorique (DT). Des densités frittées élevées (>99·3% DT) sont obtenues déjà après frittage à 1300°C pendant 2 h. Les céramiques ont une microstructure très fine avec des grains de taille moyenne de l'ordre de 0·3 μm et une distribution serrée. Le coulage centrifuge permet la préparation de céramiques massives avec une diminution de la taille des défauts ce qui entraîne une fiabilité supérieure.*

1 Introduction

According to Griffith's equation higher strength ceramic components can be achieved by either minimizing the flaw size or by increasing the fracture toughness, K_{Ic}. Higher fracture toughness values can

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be reached by utilizing the martensitic phase transformation of tetragonal to monoclinic ZrO_2. From the group of zirconia-toughened ceramics, 3 mol% Y_2O_3 TZP (tetragonal zirconia polycrystals) exhibit both small grain size ($<1\,\mu$m), as well as transformation toughening, with K_{Ic} up to $11\,\mathrm{MPa}\sqrt{\mathrm{m}}$.

Colloidal processing of Al_2O_3 has been shown to provide a route for fabricating structurally reliable ceramic bodies by decreasing the size and the number of flaws.[1,2] Centrifugal slip casting combines the advantages of wet chemical powder preparation with a highly efficient and rather stress-free densification technology to produce massive and near-net-shape ceramic parts. Isostatic pressing is the most commonly used method to produce massive ceramic parts and therefore chosen as the standard technology for comparison reasons. The wet processing route provides the possibility of breaking down agglomerates and other flaw sources by milling or by ultrasonic treatment or removing them from the slurry (decantation, sedimentation, filtration). However, one drawback of wet chemical powder consolidation by centrifugation might be the differential sedimentation due to the particle size distribution in the starting powders.

There are three different approaches to overcome this problem: Lange[3] used flocced slurries (cohesive, touching particle networks) and Velamakanni *et al.*[4] used coagulated systems (weakly attractive networks) to prevent mass segregation during centrifugation. In both cases the particle size distribution, as well as the phase assemblage (in biphase systems), are preset in the suspension network and are not changed during the densification step. Beylier *et al.*[5] used aqueous alumina suspensions with repulsive, non-touching networks containing high solid volume fractions (50 vol.%). They suggested that mass segregation could be avoided by hindered sedimentation in their high solid content slurries.

The drawbacks of the first two methods are that flocculated and coagulated systems lead to green bodies with rather open structures and lower green densities which need higher sintering temperatures to approach theoretical density. Suspensions with repulsive, non-touching particle networks lead to bodies with high green densities which show good sintering behaviour and reach high final densities at much lower sintering temperatures.

No studies of centrifugal slip casting of TZP slurries with high solids loadings are known to be reported in the literature.

The aim of the present study was to investigate the quality of centrifugal slip cast zirconia (TZP) in comparison to isostatically pressed TZP. Colloidal stability, viscosity, green and sintered densities, sintering temperature, grain size and fracture toughness were studied.

2 Experimental Procedure

A polycrystalline tetragonal zirconia powder containing 3 mol% yttria (SY Ultra 5·2, Z-Tech Pty Ltd, RG12 5BH, UK) was used throughout the entire study. Chemical analysis revealed an yttria content of 51 wt%, with the following major impurities (in ppm): 21 Fe_2O_3, 12 SiO_2, 75 TiO_2, 8 Al_2O_3, 85 S and 9 CaO. The specific surface area of the powder (BET method) was 13·1 m^2/g. The average particle size, d_{50}, was 0·27 μm with 90% less than 0·58 μm (Microtrac, Leeds & Northrup, Sumneytown Pike, UK).

The colloidal stability of aqueous suspensions was studied by ESA (electrokinetic sonic amplitude) measurements (ESA 8000, Matec Applied Science, St Hopkinson, MA, USA). ESA of suspensions with 15 wt% solids content were measured in the pH range from 4 to 11 using HCl or NaOH (Merck) for titration.

Aqueous slurries of up to 42·3 vol.% TZP with a 1·6 wt% organic based deflocculant addition (Darvan C, 25 wt% NH_4PMAA (ammonium polymethacrylate), R.T. Vanderbilt Co., Norwalk, CT, USA) were prepared at a pH between 9·1 and 9·9 and subsequently dispersed by ultrasonic cavitation (Vibra Cell VC 600 (20 kHz/600 W), Sonics & Materials Inc., Danbury, USA) for 10 min, sometimes followed by an additional attrition milling for 15 min using TZP spheres 2 mm in diameter. Viscosities were measured with a rotational viscometer (Rheomat 115A, Contraves AG, CH-8052 Zürich, Switzerland) at temperatures between 25 and 30°C. Suspensions selected for casting had a pH of 9·4 to 9·8.

Slurries were centrifuged (ZK 510, Berthold Hermle GmbH & Co., D-7209 Gosheim, Germany) in 50 cm^3 polypropylene tubes at an acceleration rate of 4000 *g* for 90 min. Green bodies had a length of about 50 mm and a diameter of 27 mm. Cast specimens were dried under controlled temperature and humidity conditions and then sintered in air at temperatures of 1250, 1300 and 1350°C with 2 h holding time. The heating rate was 1°C/min. Green and sintered densities were measured by the Archimedes principle in both mercury and water. Pore size distributions in green compacts were determined by mercury porosimetry (Hg-Porosimeter 2000, Carlo Erba Strumentazione, I-Milano, Italy) with pressures up to 2000 bar. The data were analysed using the cylindrical model.

Other samples of the same material were isostatically pressed at a pressure of 350 MPa for 3 min.

Sintering temperatures for isostatically pressed samples were 1250, 1300, 1400 and 1480°C with 2 h holding time. Sintering behaviour was determined in a differential dilatometer (Typ 802, Bähr Gerätebau GmbH, D-4971 Hüllhorst, Germany), using a sapphire reference in air.

Post-sintering annealing, in order to increase the TZP grain size and therefore the toughness, was performed at 1500°C for 2, 9, 20 and 40 h. The influence of the isothermal annealing on density, microstructure and K_{Ic} was analysed. K_{Ic} measurements were performed by the Vickers indentation method (Zwick, D-7900 Ulm, Germany).

3 Results and Discussion

3.1 Suspension stability

It has been reported elsewhere[5-7] that a high solids content in the suspensions is desirable for centrifugal slip casting to avoid preferential segregation of the suspended particles with different diameters during casting. Of the same importance is a high stability of the dispersed system, e.g. high repulsive forces on the particle surfaces which prevent them from agglomeration. It is well known that the stability of suspensions increases with increasing ESA, e.g. with increasing surface charge. Therefore ESA measurements were performed in order to determine the optimal pH conditions of TZP slurries.

Figure 1 shows the ESA versus pH for a 3Y-TZP suspension. The corresponding curve of 3Y-TZP with an addition of 1·6 wt% Darvan C, based on the solids content, is also shown. Darvan C contains ammonium polymethacrylate, an anionic deflocculant which shifts the pH of the IEP (isoelectric point) from 7·7 to 4·5 and can provide dispersed suspensions with high solids loadings.[8,9] As a consequence the optimal pH range for slurry preparation is around pH 4·5 for pure 3Y-TZP and with deflocculant addition at pH 8·5–10. The solids content in the suspension could be increased from 34 vol.% at pH 4·3 to 42·3 vol.% at pH 9·7 without changing the viscosity by using 1·6 wt% Darvan C as a dispersing agent. At the same time the green density of the centrifuged samples increased from 49·2% TD at pH 4·3 to 57% TD at pH 9·7.

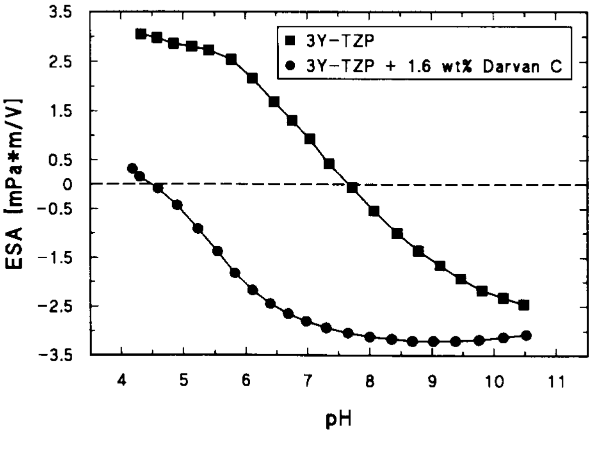


Fig. 1. ESA versus pH of 3 mol% Y_2O_3 TZP.

3.2 Viscosity

Low viscosity (≤0·5 Pas) at high solids loading is an important feature of slurries for centrifugal slip casting. The handling (mould filling) is easier and high stability is provided. Viscosity is strongly dependent on the solids content and the pH value of the suspension. The lower the viscosity at a constant solids content, the higher the stability of the dispersed system.

To optimize the stability conditions for a given slurry, viscosity measurements within a pH range around the best value pointed out by ESA measurements were performed. Figure 2 shows viscosities of TZP suspensions with a 1·6 wt% addition of Darvan C as a function of the suspension pH and solids content. For slurries with lower solids content (34 vol.%) the viscosity values are much lower and the minimum (around 0·03 Pas) covers a larger pH range.[8,9] If viscosity is too low, the interparticle distance becomes too large, resulting in separated sedimentation. Therefore the solids content was increased to 42·3 vol.%. The pH range for minimum slurry viscosity with 42·3 vol.% solids loading (0·34 Pas) is between pH 9·5 and 9·8. All high solids content slurries showed slightly pseudoplastic behaviour.

3.3 Homogenization

Homogenization treatments during slurry preparation are shown in Table 1. To lower the viscosity slightly and in order to avoid partial flocculation during centrifugation the solids content was reduced to 41 vol.% and the suspension pH was adjusted to 9·8 in the case of TZP 3 and TZP 4. Mixing of the initial suspensions was performed by ultrasonic cavitation

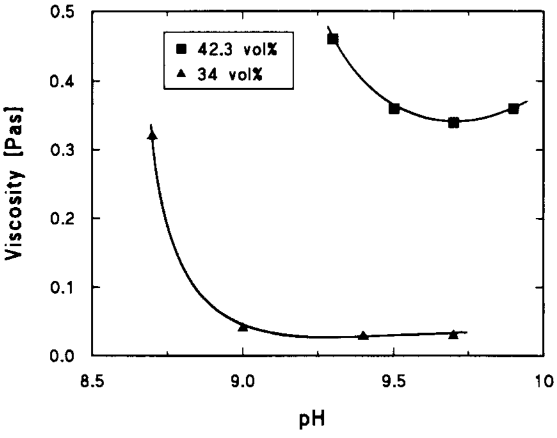


Fig. 2. Viscosity versus pH for slurries with a 1·6 wt% addition of Darvan C.

Table 1. Preparation conditions of the samples

Sample	*Vol.%*	*pH*	*Darvan C (wt%)*	*Homogenization*	*Densification*
TZP 1	34	4·3	0	10 min ultrasonic treatment	4 000 *g*/90 min
TZP 2	42·3	9·7	1·6	10 min ultrasonic treatment	4 000 *g*/90 min
TZP 3	41	9·8	1·6	10 min ultrasonic treatment	4 000 *g*/90 min
TZP 4	41	9·8	1·6	10 min ultrasonic treatment + 15 min attrition milled	4 000 *g*/90 min
TZP iso					350 MPa/3 min

Table 2. Densities at different temperatures with 2 h holding time in % of theoretical density (TD: 6·09 g/cm^3)

Sample	*Green density*	*1 250°C*	*1 300°C*	*1 350°C*	*1 400°C*	*1 480°C*	*1 500°C*
TZP 1	49·2		93·7				
TZP 2	57	98·2	99·3	99·2			
TZP 3	55·8		99·3				
TZP 4	55·7	97·3	99·8	99·2			98·5
TZP iso	53·6	84·7	94·1	96·7	98·5	99·8	98·9

(US) for 10 min alone or with additional attrition milling (Att) for 15 min (TZP 4) to investigate the influence of a combination of different homogenization methods.

3.4 Green density and porosity

Green densities and sintered densities for different sintering temperatures are shown in Table 2. All centrifugally cast samples except TZP 1 have higher green densities than the isostatically pressed sample. Green densities of samples from high volume content slurries do not differ much and are as high as 57% TD. Slurry stabilization only by pH (i.e. in the acidic region) obviously leads to a more open structure in the densified part than slurry stabilization by addition of a dispersing agent. Since TZP 1 slurries were made at a pH below 4 and subsequently adjusted to pH 4, Y^{3+} was released into the solution. Owing to the increase of ions the suspension got into a coagulated state which after casting shows a poorer particle packing than samples from well-dispersed and stabilized suspensions (TZP 2,3,4).

Pore size distributions of all green compacts are shown in Fig. 3. The particle packing of the wet processed samples is much better than that of the dry processed, isostatically pressed sample (53·6% TD) which shows a large amount of pores with a radius greater than 40 nm ($d_{50} = 32$ nm).

TZP 2 and TZP 3 are homogenized in the pH range of 9–10 using Darvan C by ultrasonication only and show a narrower pore size distribution as well as a smaller r_{50} value (19 and 20 nm) than TZP 4 (24 nm) which underwent additional attrition milling. So no further increase in green density and porosity properties was achieved by this second homogenization procedure. The pores in TZP 4 are larger but the total number of pores is smaller than in TZP 2 and TZP 3, since the amount of porosity is the same in all cast samples (*c.* 43 vol.%), except for TZP 1.

3.5 Sintering

Relative densities versus sintering temperature from sintering runs in the dilatometer at a constant heating rate of 5 K/min are shown in Fig. 4. All wet processed samples, except TZP 1, start from higher green densities and reach higher final densities at lower temperatures (including TZP 1) than the dry processed sample. The total linear shrinkage was 21·1%, 17·0%, 17·1%, 17·7% and 18% for TZP 1, 2, 3, 4 and iso, respectively. TZP iso does not show a larger shrinkage because of the rather low final density.

From Fig. 5 it appears that the maximum shrinkage rate occurs at lower temperatures

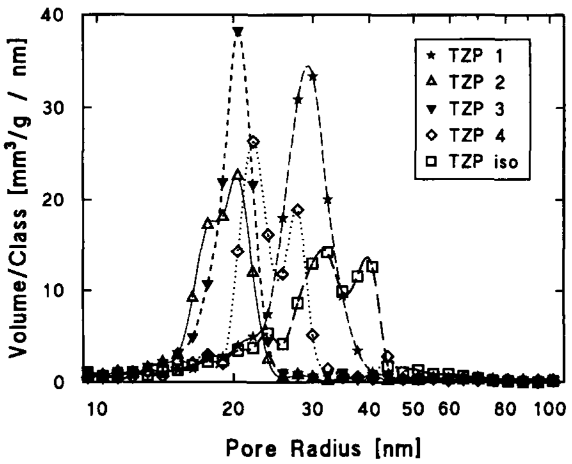


Fig. 3. Pore radius frequency distribution in centrifugally cast and isostatically pressed compacts.

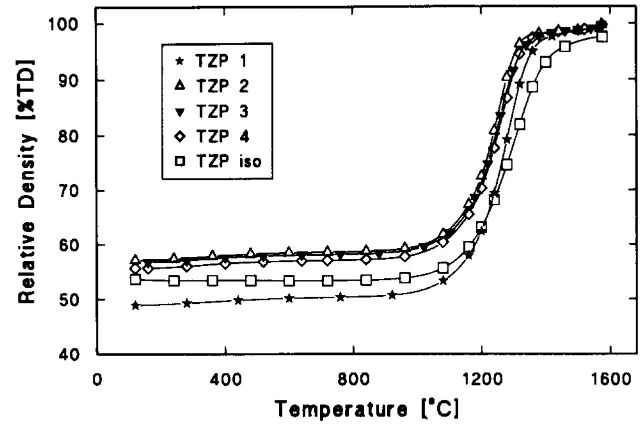


Fig. 4. Relative densities for constant heating rate (5 K/min) sintering (dilatometer).

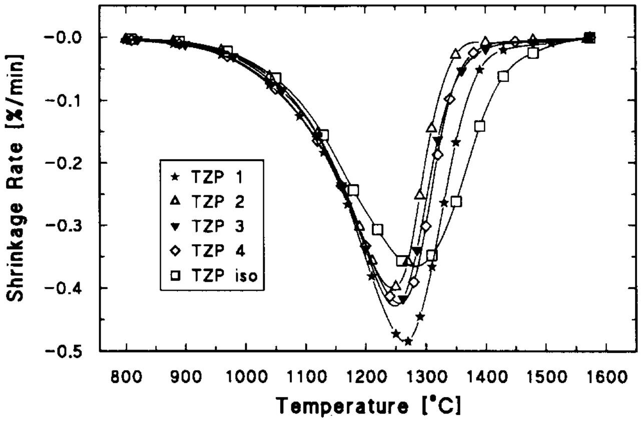


Fig. 5. Shrinkage rates during constant heating rate (5 K/min) sintering (dilatometer).

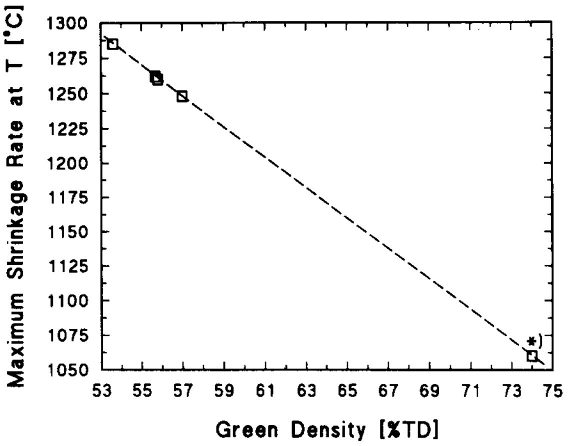


Fig. 6. Temperature of maximum shrinkage rate versus green density. *) Estimated from Ref. 1: 74% TD and 99·5% TD at 1100°C/1 h.

(20–30°C) for compacts with higher green densities. In the case of the dry pressed sample a much larger sinter interval is needed to close the structure than is needed for all wet processed samples. The large influence of green compact porosity and pore size distribution on sintering kinetics has already been noted by Roosen & Bowen[10] for Al_2O_3 and by Allemann *et al.*[11] for TZP.

The correlation between green density and temperature of maximum shrinkage rate is shown in Fig. 6. The results imply a linear relation between the green density and the maximum shrinkage rate.

Sintered densities from individual sintering runs of green compacts (of cylindrical dimensions $d = 2{\cdot}5\,cm$, $h = 4{\cdot}5\,cm$) for all specimens as well as the maximum sintering temperatures are shown in Table 2. The centrifugally cast samples sinter to >99% TD at lower temperatures than the isostatically pressed sample. TZP 2 achieved highest density between 1250 and 1300°C. A further increase of the sintering temperatures leads to a loss in sintered densities. This is a result of swelling in the final stage of sintering, as reported elsewhere.[12]

It is well known that larger pores disappear or are only reduced to an equilibrium size at higher temperatures and at a lower speed than small pores.[13] The finer the pores and the narrower the pore size distribution, the faster and earlier ceramic parts will sinter to maximum density.[1] Hence it is very important to reach a high green density as well as a narrow pore size distribution in order to achieve good sintering behaviour. Centrifugal slip casting of high solids content slurries provides ceramic parts which have high densities and narrow pore size distributions and thus show a good sintering behaviour.

3.6 Microstructure and K_{Ic} of dense TZP

The microstructure, average grain size, fracture toughness and hardness of the samples TZP 4 and TZP iso were investigated. As can be seen from Table 3 the mean grain size of the centrifugally cast sample is 0·3 μm and that of the isopressed sample of same density 0·5 μm. K_{Ic} values were found to be 6·5 $MPa\sqrt{m}$ for the wet processed and 5·9 $MPa\sqrt{m}$ for the dry processed sample. Hardness values of samples from both processing methods were in the

Table 3. Microstructure, mean grain size, fracture toughness and hardness after sintering at T_{max} for 2 h

Sample	T_{max} (°C)	*% TD*	*Mean grain size* (μm)	K_{Ic} ($MPa\sqrt{m}$)	*Hardness* (*GPa*)
TZP 2	1 300	99·3	0·3		
TZP 4	1 300	99·8	0·3	6·5 ± 0·3	13·3 ± 0·16
TZP iso	1 480	99·8	0·5	5·9 ± 0·2	13·1 ± 0·19

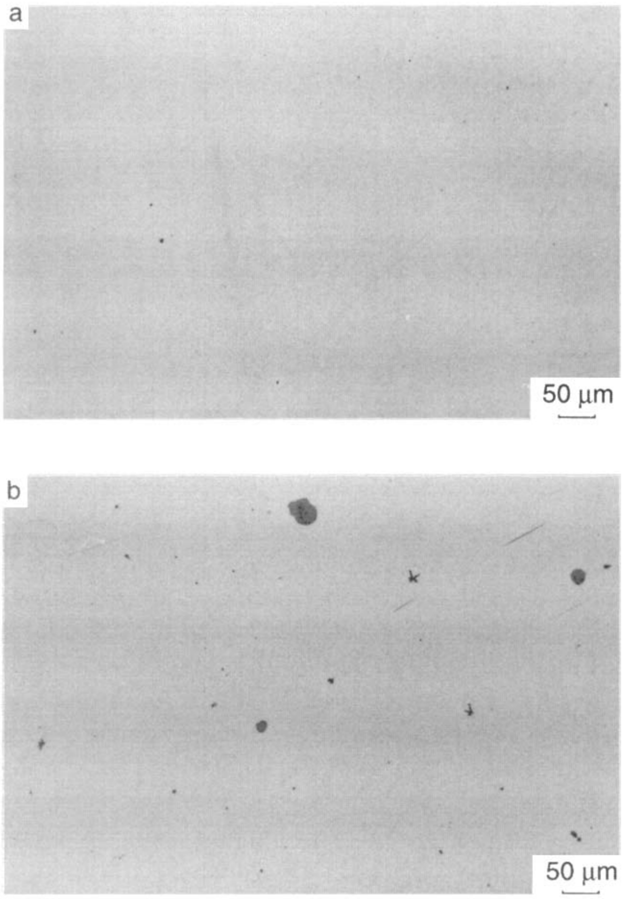
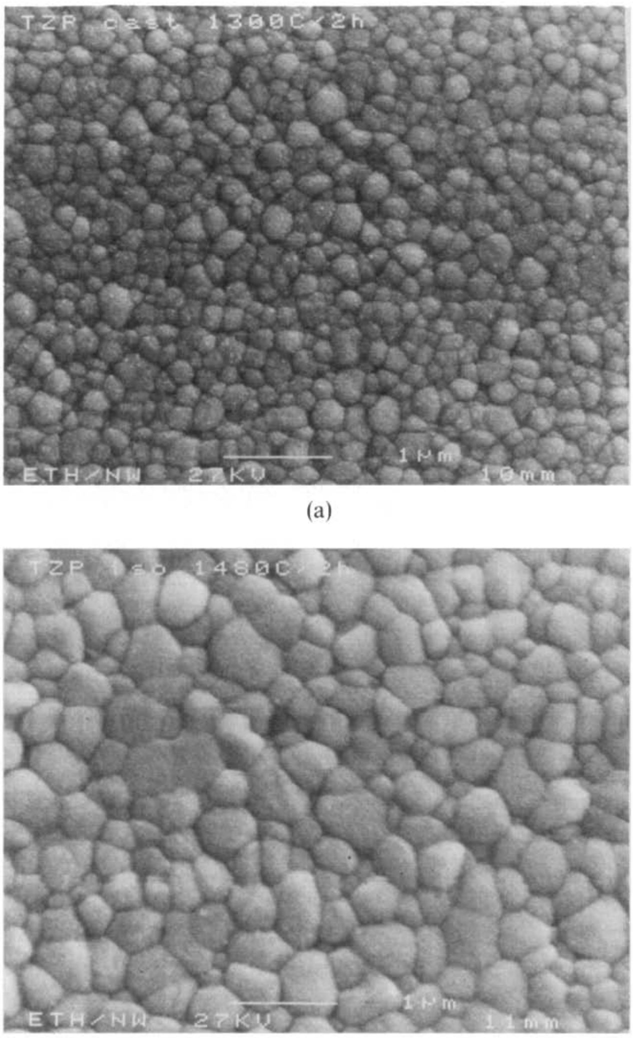


Fig. 7. Polished surfaces of cross-sections of sintered 3Y-TZP (LM): (a) centrifugally cast (TZP 2); (b) isostatically pressed (TZP iso).

same range of 13·2 GPa. The physical properties of dense centrifuged TZP do not differ from those of isopressed TZP although there is a drastic difference in sintering temperature (180°C) and therefore in average grain size.

The amount and size of flaws in centrifugally cast samples is smaller than in isostatically pressed samples. Typical microstructure overviews are shown in Fig. 7. Dark edged spots are pores induced by not totally pressed agglomerates and the larger (up to 50 μm) round dark spots are regions of lower density (agglomerates) which show a different light diffraction than the rest of the densified material. The microstructure of a dense centrifugally cast sample is finer and more homogeneous than that of isostatically pressed samples, as can be seen from the micrographs in Fig. 8. Grain size analysis revealed a smaller mean intercept and a narrower distribution for the centrifuged sample than for the isostatically pressed one, as can be seen from Fig. 9. Moreno *et al.*[14] reported that for TZP up to 1400°C no significant increase of average grain size could be observed and that the grain size increased with sintering time at temperatures higher than 1400°C. This effect is observed in the present study, since colloidal processing provides a mechanism for breaking up the agglomerates and avoiding large flaws and thus leading to better particle packing. Therefore lower sintering temperatures (1300°C) and less grain growth occurs. Wet processing is responsible for both the difference in average grain size and the difference in flaw occurrence.



(a)

(b)

Fig. 8. Microstructures of sintered 3Y-TZP (SEM): (a) centrifugally cast (TZP 2); (b) isostatically pressed (TZP iso).

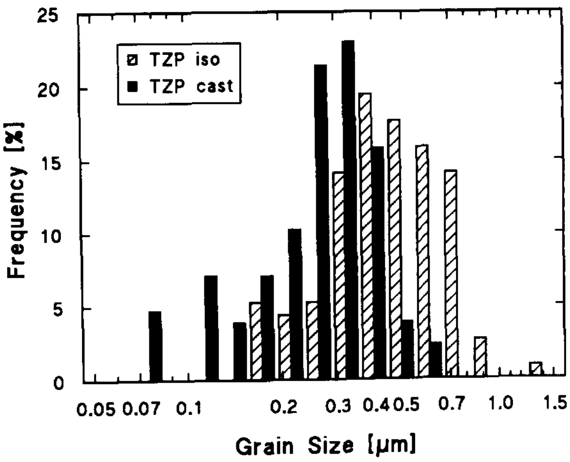


Fig. 9. Grain size distributions of centrifugally cast and isostatically pressed TZP.

Table 4. Density (D), fracture toughness (K_{Ic}) and hardness (GPa) after post-sintering heat treatment

T/hold (°C/h)	*TZP 4*			*TZP iso*		
	D (% TD)	*K_{Ic} (MPa$\sqrt{m}$)*	*Hardness (GPa)*	*D (% TD)*	*K_{Ic} (MPa$\sqrt{m}$)*	*Hardness (GPa)*
1 300/2	99·7	6·45	13·3	94·1	6·05	13·0
1 480/2				99·8	5·92	13·1
1 500/2	98·5	6·44	12·7	98·9	5·85	12·5
1 500/9	98·4	6·58	12·1	98·7	6·57	12·3
1 500/20	97·9	10·13	11·4	98·3	11·27	12·0
1 500/40	97·5	13·21	11·1	98·2	12·03	11·6

3.7 Post-sintering heat treatments

The use of post-sintering heat treatment at 1500°C for up to 40 h made it possible to increase the toughness values of both wet and dry processed samples to 13·21 MPa$\sqrt{m}$ and 12·03 MPa$\sqrt{m}$, respectively (Table 4). At the same time the density decreased for the cast sample to 97·5% TD and for the pressed sample to 98·2% TD. At this temperature the centrifugally cast sample, which is already oversintered, shows a significant loss in density. This might be due to C inclusions in grain boundaries which produce more of a bloating effect the earlier the ceramic part starts to close its structure during sintering. Since the structure is closed below 1300°C for the centrifugally cast samples, the loss of density upon post-sintering heat treatment at 1500°C is more evident than for the isopressed samples. This swelling behaviour was studied separately and the results will be reported elsewhere.[12] The dependence of the average grain size during post-sintering heat treatment was not determined owing to the formation of cubic grains at longer annealing times.

4 Conclusions

Centrifugal slip casting of high solids loading suspensions provides a route to produce massive and near-net-shape TZP ceramic parts which show better particle packing than isostatically pressed TZP. The number and size of flaws and pores is much smaller and as a consequence high green densities, as well as high sintered densities are achieved. Centrifugally cast parts from optimized suspensions reach almost theoretical density (99·8% TD) at temperatures as low as 1300°C. Centrifugally cast sintered compacts show a finer and more homogeneous microstructure than isostatically pressed TZP. After post-sintering annealing, fracture toughness could be obtained of up to 13 MPa$\sqrt{m}$. Thereby centrifugally cast materials exhibited narrower grain size distributions than dry isostatically pressed materials.

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