Journal of the European Ceramic Society 22 (2002) 1801–1809

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Sinter-HIP of α-alumina powders with sub-micron grain sizes

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Received 1 August 2001; received in revised form 12 November 2001; accepted 17 November 2001

Abstract

The densification behaviour of different nanoscale pure alumina powders by sinter-HIP to obtain specimens with sub-micron grain size has been investigated. The sinterability of these powders to determine the minimum temperature to obtain close porosity has been determined by dilatometry and by pressureless sintering in air. A good correspondence between minimum temperature to obtain closed porosity and minimum temperature to obtain full density by sinter-HIP has been determined. Different green performing routes has been analysed, uniaxial pressing and colloidal pressure filtration, being the green density obtained of 56% TD and 62% TD respectively. From the powders analysed Taimicron TM-DAR is the powder with its maximum sinterability and can be fully densified by sinter-HIP at temperature as low as 1250 °C and 150 MPa. The specimens obtained at the lowest temperature presented a grain size of 0.45 μm, a hardness of 23–24 GPa and fracture toughness $K_{IC} = 3.5$ MPa $m^{1/2}$.

Keywords: Al_2O_3; Grain size; Hardness; HIP; Porosity; Sinter-HIP

1. Introduction

Much attention has been focussed to obtain alumina ceramics with submicron microstructures in order to improve their mechanical properties. For this purpose very fine and high purity powders are required. In order to maintain the submicron scale grain size by pressureless sintering, it is necessary to apply powder processing techniques like cold isostatic pressing[1–5] (CIP), pressure filtration,[1–3] or gelcasting,[2,3] which lead to high density green bodies with good homogeneity of compaction and that allows to attain full density at low temperatures. Additionally, the properties of a ceramic material are mainly limited by the presence of various types of defects.[6] Many of those defects arise from agglomerates present in the early stage of the processing which lead to packing inhomogenity during the forming process. Sintering only tends to increase the severity of flaws already present in the green body. Thus, it is very important to make a green body or preform with a reduced density of defects. Krell et al.,[1–3] using a very fine commercial powder, obtained relative densities of 99.2% TD and grain sizes of 0.5 μm, by pressureless sintering at 1300 °C using preforms prepared by gelcasting, and at 1350 °C using preforms prepared by pressure filtration. Remarkably very high strength (3-point bending), 800–900 MPa for ground samples and 1300 MPa for polished samples, was reported. In these investigations, the high strength was associated with a low flaw "density" (defined by the frequency and size of inherent defects) that decreased to a few percentage of the flaw density observed in conventional aluminas. The number and size of agglomerates can be reduced by dispersing particles in a liquid medium, using the so-called colloidal processing. Colloidally treated slurries should not be dried before consolidation. Drying causes the spontaneous reformation of agglomerates.[6,7] Consolidated shapes, therefore, must be formed from the slurries themselves. Pressure filtration or filter pressing is such a way to consolidate ceramic slurries. Forming by pressure filtration is characterised by die pressing of a concentrated suspension (slurry) while the liquid is removed by filtration. The major advantage of pressure filtration is that it allows

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0955-2219/02/$ - see front matter
PII: S0955-2219(01)00510-6

the powder particles to be kept in a liquid dispersing medium right up to the point of particle-particle contact during the consolidation step. Since no drying stage is necessary prior to consolidation, the well-dispersed state of slurry is maintained.

Very fast densification at very low temperature by plasma activated sintering (PAS)[8–10] and spark plasma sintering (SPS)[11,12] has been recently developed. In these techniques a direct pulse current is applied for heating, which implies very fast heating rates, promoting matter transfer and make rapid densification of the powder compact or loose powder. Full density can be obtained at temperatures as low as 1150 °C, but microstructure inhomogeneities may appear as a consequence of a temperature gradient.[13]

An alternative to pressureless sintering is hot pressing (HP) and hot isostatic pressing (HIP). It is known that pressure helps sintering and densification processes, thus allowing to reduce sintering temperature. Besides, HIP in contrast to hot pressing (HP) permits to produce components with complex and precisely defined shapes. Very high strength and small grain size alumina specimens are reported to be obtained by HIP.[14–16]

The use of HIP for alumina is generally referred to the consolidation of encapsulated green performs,[14] in order to prevent the penetration of the applied gas used to transmit pressure, or to sintering in air-plus-HIP.[15,17] A further alternative is sinter-HIP, (used for silicon nitride ceramics),[18–20] which involves the sintering of non-encapsulated green preforms during a first step at low pressure (~0.5 MPa) and once closed porosity is attained, high pressure is applied subsequently for the rest of the densification period.

In the present work the sinterability of different commercial nanoscale pure alumina powders by pressureless sintering and sinter-HIP has been studied. Although basic work has been carried out using uniaxially pressed green compacts, pressure filtration has also been used in order to decrease the sinter-HIP temperature for full density. The influence of sinter-HIP parameters on the densification behaviour has also been analysed. The resulting microstructures were examined by scanning and transmission electron microscopy, and the mechanical properties (hardness and fracture toughness) were determined by Vickers indentation technique.

2. Experimental procedure

Three different ultra fine high purity (>99.99% Al_2O_3) commercial α-aluminas powders have been investigated. Their characteristics (particle size and surface area) are presented in Table 1. Green preforms were obtained (from as-received powders) by uniaxial pressing at 75–100 MPa for all powders and by colloidal pressure filtration for the Taimicron TM-DAR powder. Fig. 1 shows this processing route for alumina preforms. The first step was the preparation of an aqueous alumina slurry. The powder was dispersed in distilled water and adjusted to pH 10 by the addition of 35 vol.% ammonia. The slurry was ball milled for 20 hours in a polyethylene bottle with Y-TZP balls as the milling media, then 1 wt.% pre-dissolved celacol were added as a binder. The mixture was continuously milled for another 4 h. The received slurry was poured into a beaker and heated at a temperature of about 150 °C on a hot plate while strongly stirred using a mechanical stirrer till a gel with appropriate consistency suitable for filter pressing obtained. It is important to keep the temperature at a level, which leaves enough water in the gel in order to avoid agglomeration during this evaporation process. The second step was filter processing.

Table 1
Characteristics of commercial pure aluminas

Powder	Particle size (μm)	Surface area (m^2/g)
Sumitomo AKP-50	0.1–0.3	10.4
Ceralox APA-0.5[a,b]	0.4	9.6
Taimicron TM-DAR[a]	0.2	14.5

[a] Powders supplied by Morgan Materials Technology Ltd.
[b] Without MgO addition.

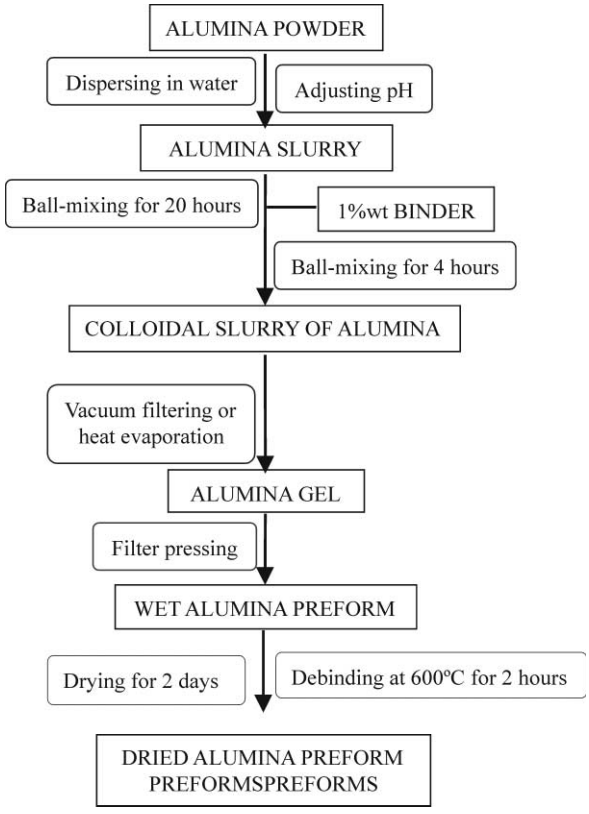


Fig. 1. Filter pressing of alumina preform from Taimicron alumina powder.

Fig. 2 gives the schematic diagram of filter press. A pressure was applied to the gel via a moving plunger. The porous filter was a partially sintered stainless-steel disc with a mean pore size of 6 μm (Porvair Technology Ltd, UK). A fine pore size filter paper (540, Whatman International Ltd, UK) was used to initiate the consolidated layer build up. During filter pressing, water in the gel moves out through the porous filter plate, and particles within the gel consolidates into preforms gradually within the inset. After consolidation, the preforms were ejected, dried in air for 2 days and finally debindered at 600 °C for 2 h at a heating rate of 3 °C/min.

The minimum temperature for obtaining closed porosity for the different powders was determined from pressureless sintering experiments carried out in static air at different temperatures, ranging from 1250–1550 °C (dwell time = 60 min). This temperature will determine the conditions of sinter-HIP experiments (for instance, the temperature at which pressure can be applied). Additionally, the sinterability of the different powders has been studied by dilatometry at 1500 °C under Ar at different heating rates: 10–100 °C/min.

Sinter-HIP experiments were performed using an ASEA-HIP equipment (QIH-6) under Ar at temperatures between 1250 and 1500 °C and pressures between 20 and 150 MPa. The green compacts were introduced in an alumina powder bed to minimise possible reactions with the graphite heating element.

Fig. 3 shows a scheme of the sinter-HIP cycle. In these experiments a pressure of 0.5 MPa was maintained constant during the whole heating period (from room temperature to the sintering temperature, T_s). After a dwell time (t_i) at this temperature, the pressure is subsequently increased and finally temperature and pressure can be maintained during a constant period of time (t_d). The influence of these variables (and the heating rate) on the density has been analysed.

Density was measured by immersion using the Archimedes principle. Microstructures were observed from fractured surfaces by SEM (Philips XL30-CP) and from thin foils by TEM (Philips CM12), which were prepared by ion-beam milling using a Gatan precision ion polishing system (PIPS).

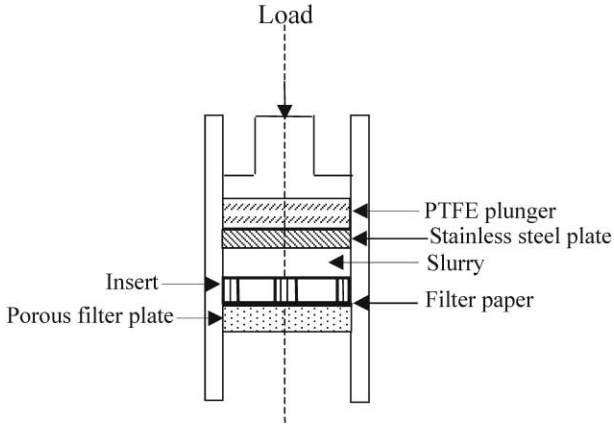


Fig. 2. Schematic diagram of pressure filtration.

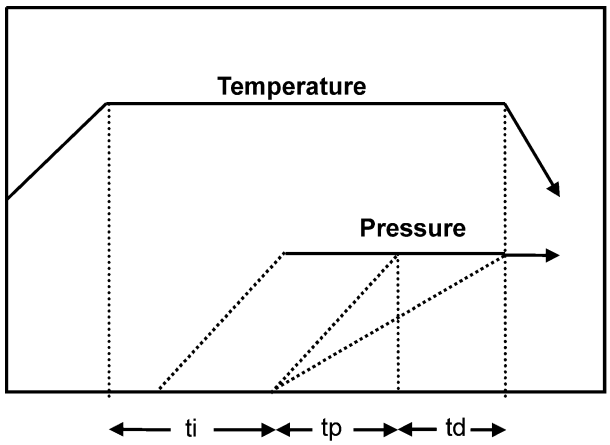


Fig. 3. Sinter-HIP scheme.

The grain size (mean linear intercept multiplied by 1.77, according to the tetrakaidecahedron geometry) was measured from TEM micrographs. Hardness (HV) and fracture toughness (K_{IC}) were measured on polished surfaces (up to 1 μm finish), using a Vickers hardness tester. For the determination of both magnitudes a load of 50 N was applied and K_{IC} values were calculated using the equation given by Anstis et al.[21]

3. Results and discussion

3.1. Pressure filtration of performs

Pressure filtration is considered to be an advantageous shaping method.[7] As is well known, agglomerates decrease powder sinterability and produce cracklike voids during densification, thus degrading the properties of ceramics. Generally, the pressure filtration process offers the possibility of avoiding uncontrolled agglomeration by processing the powder in a liquid suspension without any drying step before shaping. It is well recognised that the particle arrangement is influenced by the applied pressure. Generally, particle packing density increases with pressure. However, the applied pressure can not be too high, otherwise the preform would be cracked due to excessive stresses formed within it. It was found that the immediate application of high pressures would cause the gel to move out through the seam between the edge of the plunger and the wall of the die, especially when the fit is not adequate. This problem can be overcome by first applying a lower pressure to produce a substantial consolidated compact and then increasing the pressure to the desired one to complete the consolidation. Fig. 4 shows the effect of applied pressure during filter pressing on the relative density of

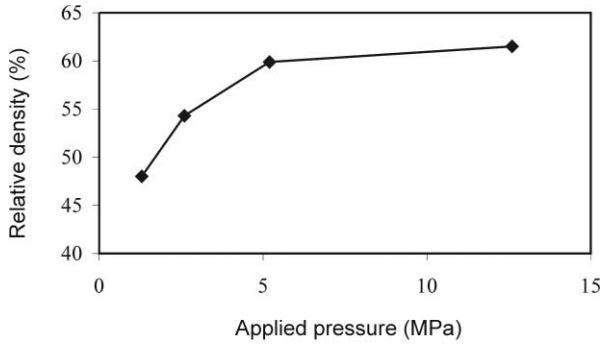


Fig. 4. Relative density of Taimicron alumina preform vs applied pressure during filter pressing.

Taimicron alumina preforms. It can be seen that the density increased as the pressure increased and when the pressure was 13 MPa, preforms with a relative density of 62% can be obtained.

3.2. Pressureless sintering study

Green compacts uniaxially pressed at 75 MPa were used for this study. The green density for AKP50 was lower (53% TD) than for Taimicron and Ceralox powders (56% TD).

The densification and shrinkage rate curves obtained by dilatometry at 10 and 30 °C/min for the different powders are presented in Figs. 5 and 6 respectively. The temperature for the maximum shrinkage rate for the different powders and conditions is also included in these figures.

As expected and according to the values presented in Table 1, the sinterability of the different powders depends on their characteristics, basically on the surface area. Sumitomo AKP50 and Taimicron TM-DAR have approximately the same particle size (0.2 μm) but the latter one has a much higher surface area (14.5 m^2/g).

Taimicron TM-DAR is the powder with the highest sinterability, and the maximum shrinkage rate is attained at the lowest temperature. Fig. 6 shows that the maximum shrinkage rate is displaced towards higher temperatures for higher heating rates, attaining higher values.

This effect can also be observed in Fig. 7, in which the shrinkage rate for AKP50 powder during heating (up to 1500 °C) is presented as a function of time (Fig. 7a) and temperature (Fig. 7b) for different heating rates (10–100 °C/min). As shown in this figure rapid densification can take place at the highest heating rate. As the heating rate is increasing, the specimens are less time at intermediate temperatures, less densification occurs at these temperatures, leading to a higher driving force for densification, thus avoiding the possibility of exaggerated grain growth. Although not shown in this figure, the final density increases slightly with the heating rate, in agreement with the results reported for fast firing.[11,12]

The relative bulk densities obtained by pressureless sintering in air (heating rate = 30 °C/min) of the different

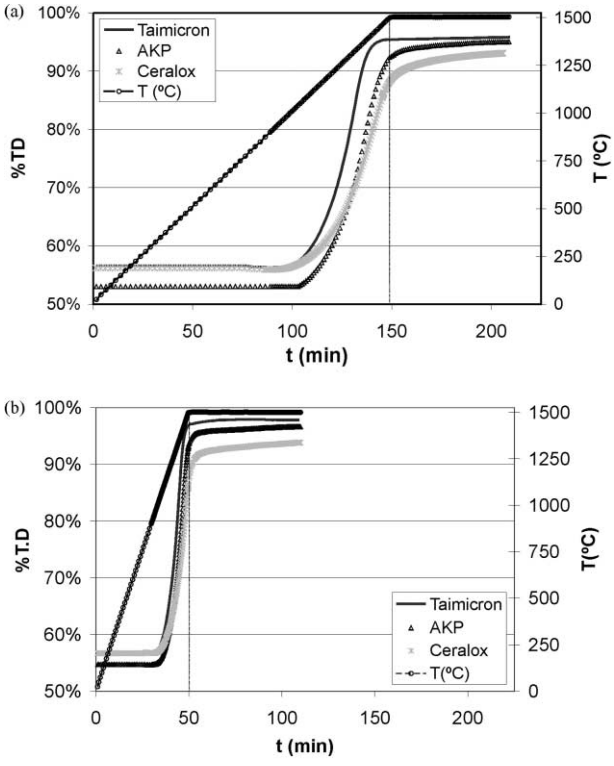


Fig. 5. Densification curves obtained in the dilatometer at (a) 10 and (b) 30 °C/min.

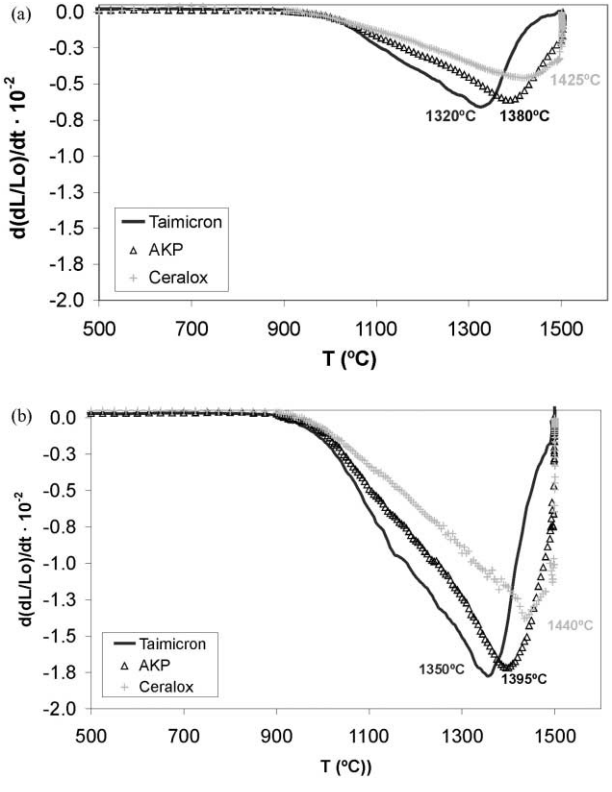


Fig. 6. Shrinkage rate curves at (a) 10 and (b) 30 °C/min.

powders as a function of the temperature are shown in Fig. 8. In the figure the dotted line represents the minimum density for closed porosity (92%) it has been considered

As shown in this figure, at 1500 °C all powders achieve the same density (~98% TD), however, at $T<1450$ °C the final density depends on the powder characteristics, and the tendency is the same to that found in the dilatometry analysis.

From this figure the lowest temperature at which closed porosity is obtained can be determined, being the lowest for Taimicron (1275–1300 °C, depending on the heating rate).

Although there was a limitation in the maximum heating rate for the pressureless sintering experiments (30 °C/min), also in this case a dependence of the final density with the heating rate has been found. Table 2 shows the density and the minimum temperature to obtain closed porosity corresponding to the different powders pressureless sintered at 1350 °C and 10 or 30 °C/min. It must be noticed that these temperatures can be slightly lower (<25 °C) for pressureless sintering performed at higher heating rates.

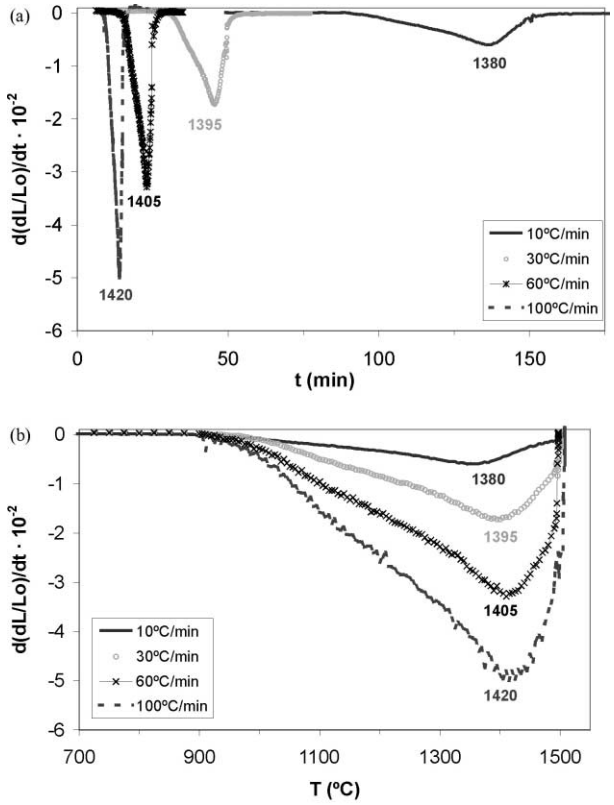


Fig. 7. Shrinkage rate for the different heating rates (for AKP50 powder) as a function of (a) time and (b) temperature.

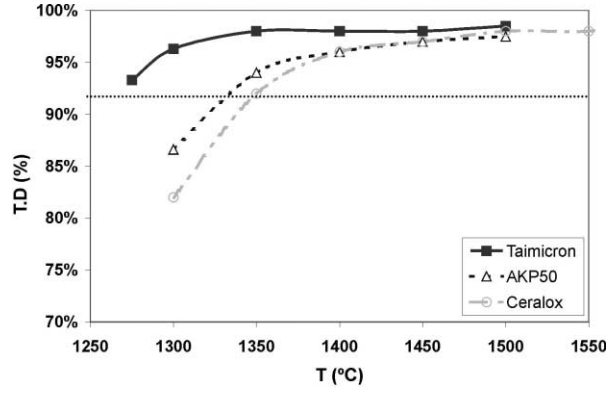


Fig. 8. Bulk density by pressureless sintering at different temperatures.

3.3. Sinter-HIP

The comparison between the densification by sinter-HIP of the different alumina powders has been carried out using a standard sinter-HIP cycle (cycle I) with the following conditions (Fig. 3): $t_i = 30$ min; $t_d = 30$ min; heating rate = 10 °C/min. The pressurisation rate was ~3 MPa/min (the maximum that the HIP equipment can stand). In all cases the applied pressure was 150 MPa. Uniaxially pressed green compacts were used for this study.

Fig. 9 shows that the simultaneous application of pressure and temperature tends to achieve full density at temperatures lower than 1400 °C for all the powders analysed. Below this temperature a good equivalence between the results obtained by pressureless sintering in air (and in the dilatometer) with the sinter-HIP results

Table 2
Density at 1350 °C (dwell time = 60 min) and minimum temperature necessary for closed porosity in air, at 10 or 30 °C/min

Powder	10 °C/min		30 °C/min	
	Density (T.D%)	T_{min} (°C)	Density (T.D%)	T_{min} (°C)
Taimicron	97.2	1300	97.8	1275
AKP50	93.9	1350	95.0	1325
Ceralox	92.5	1375	94.3	1350

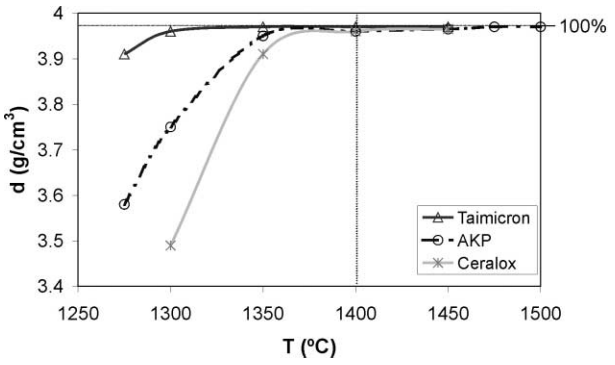


Fig. 9. Evolution of the bulk density with temperature for sinter-HIP cycle I.

has been found. In these terms the minimum temperature for full density by sinter-HIP is nearly the same as the minimum temperature for closed porosity determined by pressureless sintering in air. This temperature also depends on the powder characteristics. Table 3 compares both temperatures for the different powders.

It is important to point out again the Taimicron TM-DAR powder, whose minimum temperature for full density is remarkably low: 1300 °C (under conditions of cycle I).

In order to optimise the sinter-HIP cycles, and to reduce the total cycle time, the influence of the different parameters such as the starting pressurisation time (t_i), the dwell time at pressure (t_d) and the heating rate were studied.

From pressureless sintering studies the beneficial effect of heating rate in the densification had been determined. Therefore, subsequent experiments were carried out at the maximum heating rate that the HIP equipment stands for (30 °C/min).

Table 4 shows, for the AKP50 powder, the influence of t_i, in which the bulk density (%TD.) for different t_i (before and after the pressurisation) is included. It must be noticed that in all cases, before the start of pressurisation, the specimens did not contain open porosity and full density is obtained at 150 MPa. It is important to mention that in these experiments $t_d = 0$.

From these results it can be concluded that a t_i of 5 min is enough (at this temperature) to obtain closed porosity that allows to be fully dense after pressurisation.

Based on these arguments a second cycle (cycle II) was developed in order to optimise the properties of the sinter-HIPed specimens. In Fig. 10 both cycles are compared. The second one implies a considerable reduction of the cycle time, from 250 to 100 min. It is noteworthy that the final bulk density was even higher. Therefore, Taimicron powder can be fully dense at 1275 °C ($P = 150$ MPa), at which a density of 99.7% TD was obtained for a green compact.

Table 3
Minimum temperature for full density by sinter-HIP (cycle I) and for closed porosity by pressureless sintering in air

Powder	$T_{\text{sinter-HIP}}$ (°C)	T_{air} (°C)
Taimicron	1300	1300
AKP50	1350	1350
Ceralox	1400	1375

Table 4
AKP50 density (T.D%) at 1350 °C for different t_i with and without pressurisation

	$t_i = 5$ min	$t_i = 10$ min	$t_i = 30$ min	$t_i = 60$ min
$P = 0.5$ MPa	93.0	94.0	95	95.5
$P = 150$ MPa	>99	>99	>99	–
	↑		↑	
	Cycle II		Cycle I	

The influence of the green density and the method used to obtain preforms from Taimicron powder on sinter-HIP densification has also been analysed. For this purpose green compacts obtained by uniaxial pressing and filter pressed preforms prepared at The University of Birmingham were used. As shown in Table 5, the green density obtained by filter pressing (62% TD) is notably higher than that obtained by uniaxial pressing (56%), and similar to that reported by Mizuta et al.[16] using the same powder and a similar processing route. Additionally, filter pressing should help to produce a green body with an improved density distribution.

Table 5 shows the bulk density (%TD) of the sinter-HIPed specimens performed by uniaxial pressing and filter pressing. It can be seen that for the same temperature the bulk density of the sinter-HIPed filter pressed specimens, due to their higher green density and/or a more uniform density distribution, is higher, especially at the lowest temperatures. This means therefore that filter pressed preforms permits to obtain full density at temperatures as low as 1250 °C, in comparison to 1275 °C for uniaxial pressed. It is noteworthy that at this temperature the grain size is at sub-micron scale and lower than 0.5 μm. Similar HIP densities were obtained previously by other authors[16] after sintering-plus-HIP filter-pressed green compacts at similar temperatures, but for longer HIP schedule times.

The minimum sinter-HIP temperature for full density could be reduced if the green density of filter-pressed preforms was higher as it is obtained by Krell et al.[2] (66%) by pressure filtration of binder-free slurries.

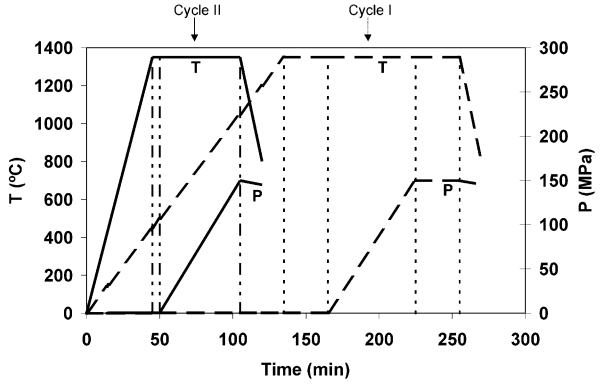
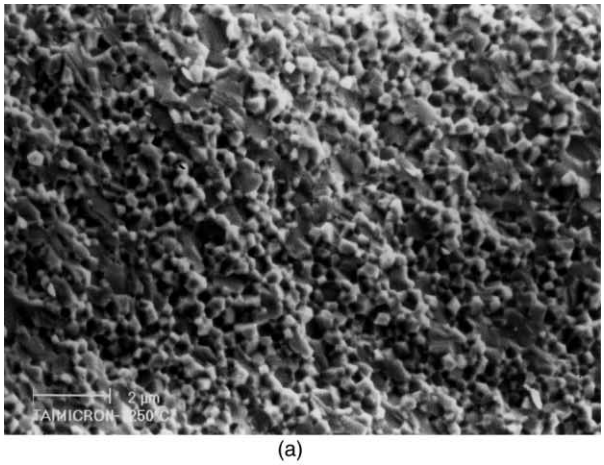


Fig. 10. Sinter-HIP cycle I (dotted line) and cycle II (solid line) schemes.

Table 5
Bulk density obtained by sinter-HIP (cycle II) for different preforms processing

Preform	1200 °C	1225 °C	1250 °C	1275 °C
Green compact (56% T.D)	95.0%	94.8%	98.3%	99.7%
Filter pressing (62% T.D)		96.0%	100%	100%



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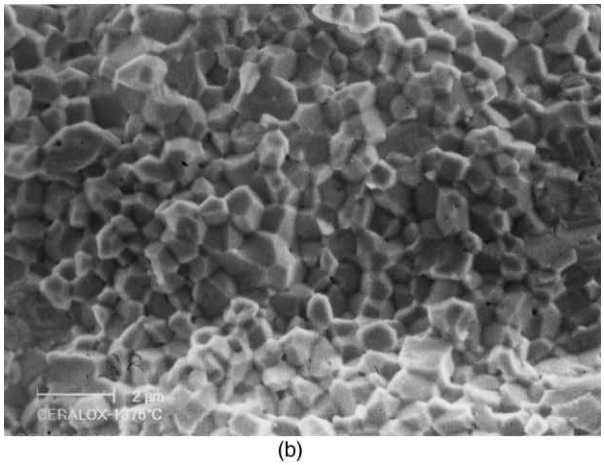

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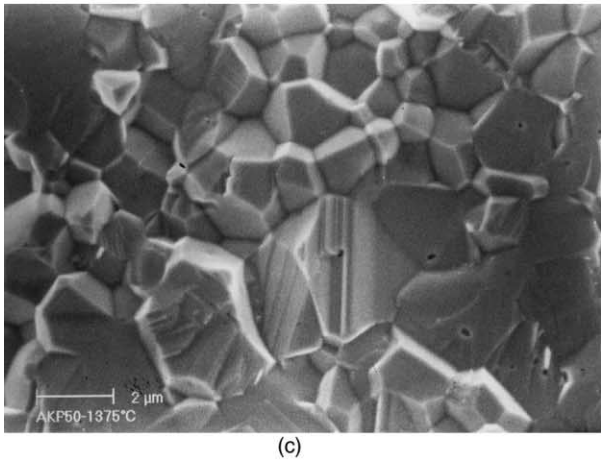
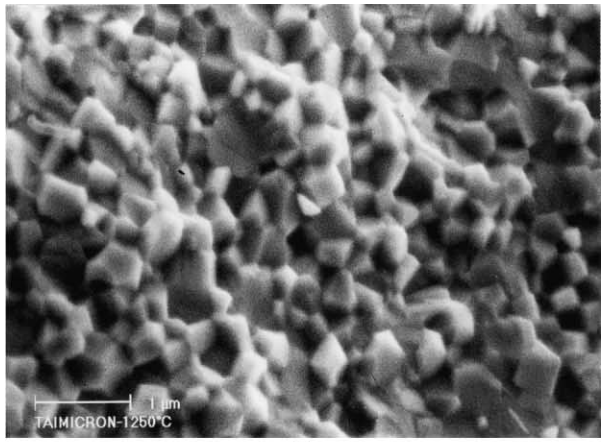


(c)

Fig. 11. SEM micrographs (fractured surfaces) of sinter-HIPed specimens. (a) Taimicron 1250 °C, (b) Ceralox 1375 °C, (c) AKP50 1375 °C.



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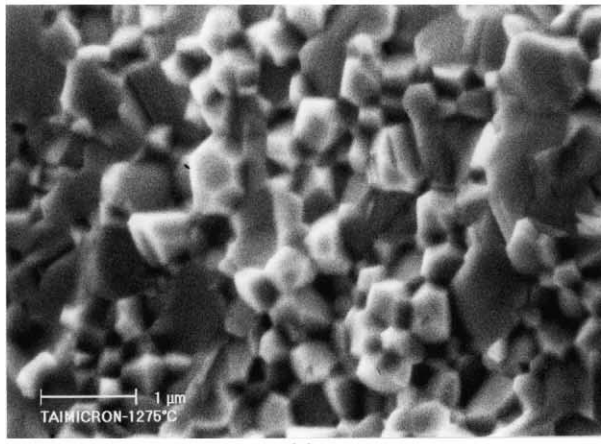

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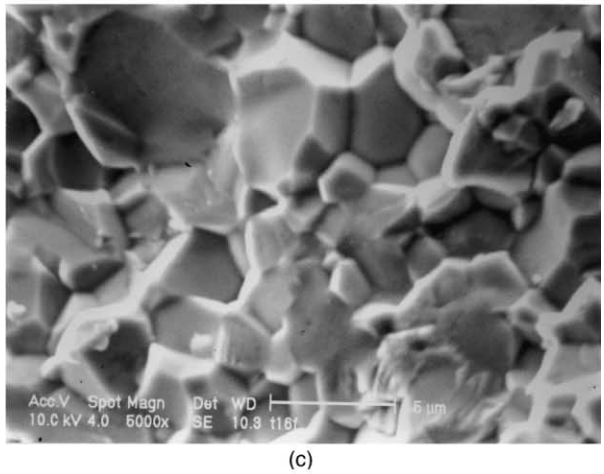

(c)

Fig. 12. SEM micrographs of Taimicron sinter-HIPed specimens at different temperatures: (a) 1250 °C, (b) 1300 °C, (c) 1400 °C.

3.4. Microstructural characterisation and mechanical properties

As mentioned before, all these powders can be fully densified by sinter-HIP at much lower temperatures than by pressureless sintering in air. Since Taimicron TM-DAR is the powder, which can be fully densified at the lowest temperature, its grain size may be expected to be the smallest. Fig. 11 compares the microstructures (from fractured surfaces by SEM) of sinter-HIPed specimens from the different powders at their lowest temperature at which full density was

obtained. It is clearly seen that in fact Taimicron sinter-HIPed specimens show the smallest grain size. The grain size of both Taimicron and Ceralox specimens are at sub-micron scale, and do not experiment appreciable grain growth, since their grain size is similar to their initial particle size (0.2 and 0.45 μm respectively). Therefore, it is noteworthy that during the pressurisation time of the sinter-HIP cycle (150 MPa, in ~50 min) no significant grain growth occurs at these temperatures.

On the other hand, AKP50 powder, whose particle size is 0.1–0.3 μm, experiments appreciable grain growth attaining a grain size close to 1.5 μm at its minimum sinter-HIP temperature for full density (1350 °C).

Additionally, as shown in Fig. 12, the Taimicron powder maintains sub-micron scale microstructures by sinter-HIP at $T < 1350$ °C. It is worth emphasising that for the same sinter-HIP temperature the grain size of the filter pressed specimens is clearly lower than that obtained by uniaxial green compacts. Besides, the grain size below this temperature is very uniform, specially specimens obtained by pressure filtration. At 1250 °C the grain size is lower than 0.5 μm, whereas at 1400 °C is of ~2 μm.

Fig. 13 compares the microstructure obtained by pressureless sintering in air at 1350 °C ($d = 98\%$TD,) and by sinter-HIPed at 1250 °C ($d = 100\%$ TD). It can be seen clearly the important reduction in grain size obtained by sinter-HIP (1.7 μm vs. 0.4 μm). It is worth emphasising that for the same HIP densities (obtained at similar temperatures) the grain size is smaller than that reported by Mizuta et al.[16] as result of the shorter HIP cycle used in our case.

Table 6 summarises the mechanical properties (hardness and fracture toughness) of Taimicron specimen sinter-HIPed at different temperatures. It is noteworthy that due to the submicron scale grain size attained below 1300 °C, the hardness is markedly very high (23–24 GPa). Although there has not found any difference between the hardness of filter-pressed and uniaxial pressed preforms when they are fully dense, as a consequence of the much lower flaw frequency of filter-pressed preforms, its strength should be much higher.[2,3]

Additionally, relationed to the grain growth that takes place from 1300 °C, the hardness decreases as the temperature increases. Moreover, the average fracture toughness determined was 3–3.5 MPa $m^{1/2}$ with no influence of temperature and grain size, in agreement with other authors.[2]

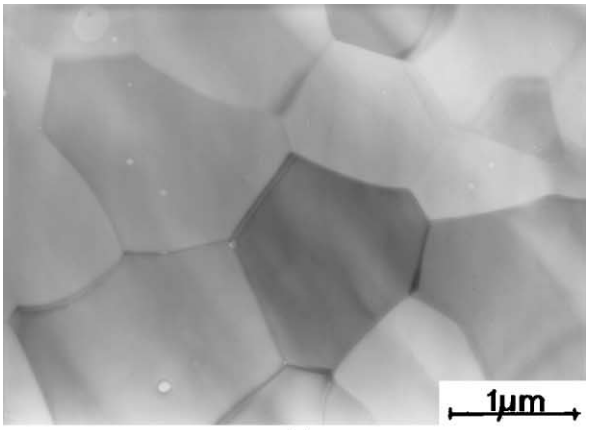

Fig. 13. TEM micrographs of Taimicron specimens: (a) pressureless sintered at 1350 °C, (b) sinter-HIPed at 1250 °C, (c) detail of (b).

Table 6
Mechanical properties of Taimicron specimens sinter-HIPed at different temperatures

T (°C)	d (g/cm^3)	HV50N (GPa)	K_{IC} (MPa m$^{1/2}$)
1250	3.90	19.9	3.4±0.3
	3.97*	23.7*	3.5±0.3*
1275	3.96	24.1	3.5±0.3
	3.97*	23.7*	3.0±0.3*
1300	3.97	24.0	3.0±0.3
1350	3.96	22.8	2.5±0.1
1375	3.97	22.6	
1400	3.97	21.4	3.5±0.4

*Filter-pressed preforms.

4. Conclusions

High purity, nanoscale alumina powder can be fully densified by sinter-HIP at very low temperatures, leading to submicron scale microstructures.

Taimicron TM-DAR is the powder with the highest sinterability, related with its highest surface area (14.5 m^2/g). A relationship between the minimum temperature to obtain close porosity and that to obtain full density by sinter-HIP has been found for the different powders.

By the correct design of die and inset, filter pressing can be used to produce homogeneous preforms with different shapes and sizes. Removing the excess water in alumina slurry by heating- evaporation can reduce the time needed for filter press, but it is important to keep enough water in the gel in order to avoid agglomeration by drying. The preform density increased as the pressure increased. When the pressure was 13 MPa, Taimicron alumina preforms with a relative density of 62% were obtained.

Uniaxial green compacts (~56%) can be fully densified by sinter-HIP at temperatures as low as 1275 °C. This temperature can be even lower (T=1250 °C) using preforms with higher green density (62%) prepared by pressure filtration.

The grain size is maintained at sub-micron scale at temperatures below 1350 °C, being lower for the filter pressed specimens (0.45 μm at 1250 °C). Pressurisation during sinter-HIP does not induce a significant grain growth.

The hardness and fracture toughens determined by Vickers Indentation are HV= 23–24 GPa, K_{IC}= 3.0–3.5 MPa $m^{1/2}$ respectively. Whereas fracture toughness is independent on the processing variables, the hardness decreases with temperature as a consequence of the grain growth.

Acknowledgements

The authors wish to acknowledge Morgan Materials Technology Ltd. for the supply of the powders, and the European Community under the "Competitive and Sustainable Growth" Programme (Contract No. G5RD-CT-1999-00052) for the financial support provided to undertake this work.

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