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Effect of pH on the dispersability of silicon carbide powders in aqueous media

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Abstract

The dispersion behaviour of commercial silicon carbide (SiC) powders of various grit sizes in deionised water was studied over the pH range 2–11. The slips were characterised for the state of their dispersion by employing the various quantitative diagnostic techniques e.g. particle size distribution, sedimentation, rheological behaviour, viscosity and zeta potential. SiC powders were found to get optimally dispersed at a pH value of 10 at which their near newtonian slips displayed a minimum in sedimentation height and viscosity and a maximum in zeta potential. In the acidic (pH = 2–7) range the slips were highly flocculated as manifested by larger sedimentation height, higher viscosity, lower zeta potential value and thixotropic, non-newtonian flow behaviour. Moderate to good dispersion conditions were observed in the alkaline range of pH = 7–10. The above results corroborate well with the casting behaviour of these slips. The density and moisture content as well as the green microstructure of the cast product bear a direct relationship to the state of aggregation of the slips induced by the alterations in the pH of the slurry. © 1999 Elsevier Science Limited and Techna S.r.l. All rights reserved

Keywords: A. Slip casting; D. Silicon carbide; Aqueous dispersion; Slip characterisation; Viscosity; Zeta potential; Sedimentation

1. Introduction

Covalent ceramic materials like silicon carbide (SiC) and silicon nitride (Si_3N_4) have now been recognised as potential candidates for high temperature structural applications in advanced heat engine parts (e.g. diesel and gas turbine engines), heat exchangers and friction and wear resistant components. They are slated to replace super alloys because of their superior mechanical (strength and hardness), chemical (oxidation and corrosion resistance) and thermal stability at high temperatures [1,2]. Their widespread application is, however, limited by the critical and complicated manufacturing technologies involved in producing strong and tough components from them. As ceramics are highly sensitive to flaws introduced during various stages of processing/fabrication [3], is necessary to make these components with as few flaws as possible in order to achieve the best of properties for a reasonable performance and service life [4,5]. In most cases, the fracture-initiating, strength-limiting flaws such as pores, voids, agglomerates or inclusions getting introduced during the green processing/forming stage result in a sintered body with inhomogeneous microstructure which would thereby affect the mechanical properties of the material [3–6]. While it is difficult to eliminate these strength limiting flaws when dry powder processing route is followed, consolidation routes involving wet processing (e.g., colloidal consolidation techniques) effectively overcome the above problems by electrostatic or steric stabilisation of the dispersions to yield defect-free, homogeneous and dense green compacts thereby leading to better final products [7–14].

The electrostatic stabilisation of the suspension is accomplished by generating a common surface charge on the particles either by adjustment of pH of the suspension or by the adsorption of charged species added in the form of appropriate electrolytes [8,13–18] in very small concentrations. Though a number of studies on colloidal processing of SiC are available [11–13,17–22] little work has been reported on achieving effective stabilisation of SiC in an aqueous medium by a simple pH control. (In this article words like slips, slurries, suspensions etc., are used interalia interchangeably to

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denote SiC powder dispersions in deionised water with decreasing solid loadings in the range of 60–5 wt%.) In the present study various slip/slurry characterisation techniques like sedimentation, rheology, viscosity and zeta potential measurements have been used for optimising the aqueous SiC slips as a function of pH and results are correlated with the characteristics of slip cast body.

2. Experimental procedures

Commercially available α-silicon carbide powders (M/s Grindwell Norton, Bangalore, India) of different grit sizes (namely G600, G800, G1000, & G1200) and from various batches were used for the studies. The powders were characterised for phase contents by X-ray diffraction (X-ray diffractometer, model PM9002, M/s Philips, Holland). The powders were dispersed in deionised water with continuous magnetic stirring and the pH of the slurry was adjusted to various values ranging from 2 to 12 using HNO_3 for the acidic and NH_4OH solution for alkaline range respectively. The particle size distributions of the powders were determined by sedimentation principle (Sedigraph 5100, M/s Micromeritics, USA) using 5 wt% solid loaded suspensions adjusted to different pH values. The rheological behaviour of SiC slips of different solid loadings (47–62 wt%) has been studied by measuring their viscosity and shear stress as a function of shear rate and pH of the slurry (Rotational viscometer, Searle type, Viscotester VT-500, M/s Haake, Germany). The zeta potential of the 20 wt% solid loaded SiC suspensions were measured as a function of pH (in the range of 2–11) via measurement of their electrophoretic mobilities (Zeta potential analyzer, model 1202, M/s Micromeritics, USA).

The sedimentation behaviour of the slips was studied as a function of time at different pH values (2–12) for 20 wt% solid loaded SiC slips. The sedimentation heights were measured from the bottom of the sedimenting test tube to the height of the solid deposited after specific intervals of time. These values when plotted as a function of time and pH of the slurry were used to arrive at the conditions of best dispersion. In another approach [21] the total height of the slips in the tube was taken as height at time t = 0 and subsequently the maximum level of turbidity (the solid sedimented plus the solid particles under dispersion) below the clear liquid as the sediment height at different time intervals.

In order to substantiate and confirm the results of slurry characterisation experiments, casting of 50 and 60 wt% SiC slips at selected pH values were performed in plaster moulds. After the pH adjustment the slip was subjected to 5 min of ultrasonication before casting. The moisture content and density of green body were determined by weight and volume measurements. The state of dispersion of SiC particles at different pH values was also examined by microstructural observation of slip cast green bodies on a scanning electron microscope (model JSM 35, M/s Jeol, Japan). The results of various experimental techniques are correlated to arrive at a suitable pH for obtaining an optimum dispersion of SiC particles in aqueous media.

3. Results and discussion

3.1. Particle size distribution

When particles are dispersed in a polar medium like water, the surface charges on the particles are known to influence their state of aggregation or dispersion. With the variations in pH, the particles either undergo flocculation or deflocculation depending upon the polarity and the extent of surface charges developed by the particles. The effect of pH on dispersion of SiC G1000/2 particles in deionised water in Fig. 1 shows high flocculation at pH = 3 with a narrow floc size distribution (10–30 μm). At pH = 5 the large agglomerates break down into smaller flocs, but considerable agglomeration (1–20 μm) still persists. At pH = 10 the particle size distribution is shifted towards finer range (0.8–15 μm) indicating complete deagglomeration and optimum dispersion. A very high pH value of 11.5 leads to considerable amounts of reagglomeration with a coarser and narrower size distribution than that at pH = 10. These results show that the pH of the medium greatly influences the dispersability of SiC powders even at a lower solid loading (5 wt%) and particle size analysis proves to be a vital experimental tool to investigate the dispersion levels of a ceramic powder.

The particle size distribution results of silicon carbide powders of various batches and grit sizes measured in deionised water at the pH value of 10 are presented in Table 1 along with solid loading and green density data

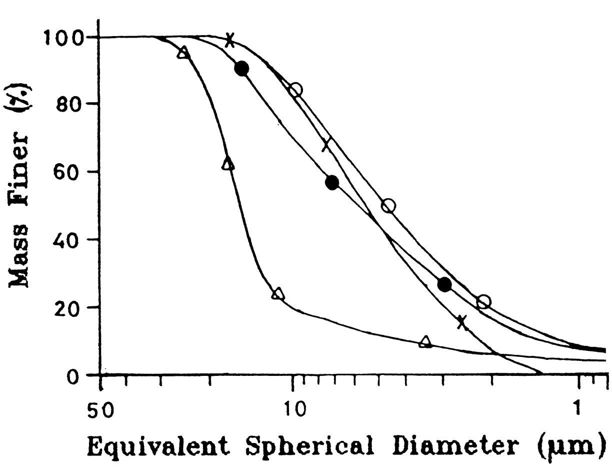

Fig. 1. Effect of pH on particle size distribution of silicon carbide G1000/2, Δ = pH 3, ● = pH 5, ○ = pH 10, X = pH 11.5.

for samples cast from the same slip (pH = 10). The results show that powders A, B and C are almost similar but A having lesser amount of fines (< 1 μm) and slightly larger amount of coarse (10–30 μm). Powders E and H have larger percentages of fines (20–25%) and smaller percentages of coarse (5–10%). While powders D, F and G have small percentage of fines (5–10%) and narrow size distributions as compared to other powders which have wider size distribution with considerable amounts of fines (15–25%). A wider particle size distribution with considerable percentage of finer particles improves the packing efficiency resulting in higher solid loaded but low viscosity slips which could in turn lead to castings having higher green density [16,23].

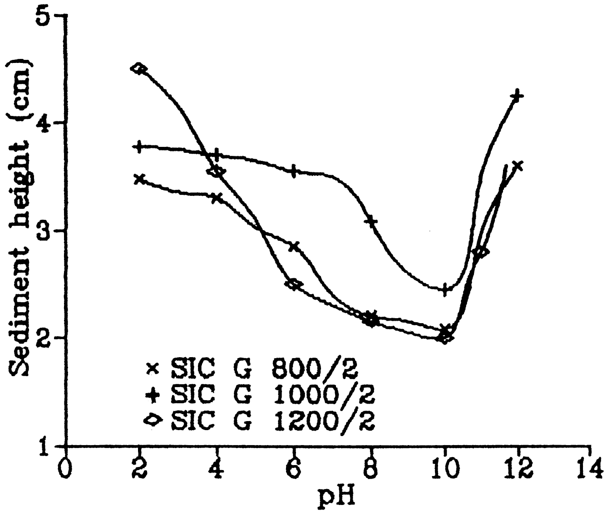

Fig. 2. Sedimentation height (24 h) plotted as a function of pH for silicon carbide powders.

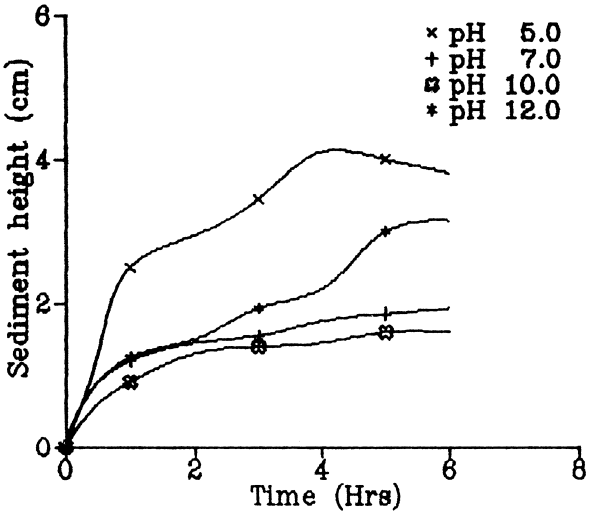

Fig. 3. Sedimentation height as a function of time for silicon carbide (G1200/2) at various pH values.

3.2. Sedimentation studies

When a ceramic powder is dispersed in a liquid medium, the particles are influenced by a variety of forces like force of gravity, drag force from the medium, brownian motion of particles etc. Assuming that the other forces are negligible or non-varying type, the ceramic particles tend to settle under the force of gravity. The particle settling rate manifested in terms of an increase of sediment height or sediment volume as a function of time is a measure of the state of dispersion and its stability. The sedimentation heights measured after 24 h for SiC powders of various grit sizes are presented in Fig. 2. A variation in the sedimentation heights as a function of pH is attributable to different levels of agglomeration or flocculation of the particles leading to different levels of packing in free space under the force of gravity. Under the conditions of good dispersion or deflocculation there is a minimum in sedimentation height since the particles settle into tightly packed sediments to give ideal packing. On the contrary, in a flocculated system, the agglomerated particles fall rapidly under gravity, (larger weight) resulting in a loosely packed sediment which leads to larger sedimentation heights. The sediment height is inversely related to the dispersability of the powder; the more dispersed the

Table 1
Particle size distribution and slip casting characteristics of silicon carbide powders of various batches

Powder type	Grit size	Average dia. (μm) Median	Modal	% Fine in the range (μm) < 1 (Fine)	1–5 (Medium)	5–10	10–30 (Coarse)	Solid loading (wt%)	Green density (%)[a]
A	G 600	5.82	8.95	7.7	36.4	31.3	24.4	71	57
B	G 800/1	4.37	7.14	15.8	39.1	27.0	16.9	71	63
C	G 800/2	5.04	8.63	14.6	35.1	29.2	20.0	75	64
D	G 800/3	5.97	7.72	5.7	35.0	41.0	18.1	65	53
E	G 1000/1	3.11	3.70	20.2	47.7	21.1	10.6	75	64
F	G 1000/2	4.84	6.54	9.3	42.1	31.6	16.7	65	53
G	G 1000/3	4.75	7.19	9.5	42.7	31.5	15.7	65	53
H	G 1200	2.55	3.59	24.5	52.8	17.9	4.5	71	58

[a] Percentage theoretical density.

powder, the smaller the sediment height. All the silicon carbide powders settled to lower sedimentation heights in the pH region 7–10 with the minimum being at the pH value of 10 showing this as the pH of optimum dispersion. The result is in accord with the earlier observations made by Freedman et al [12] for 9 wt% solid loaded aqueous SiC slips.

Usually, the stability of a slip is a function of dispersability and the time of standing of the dispersion. This effect could be seen in Fig. 3 where the sedimentation heights are plotted as a function of time at different pH values. The maxima of sedimentation heights at the pH of 5 reveals a high degree of flocculation at all times for this pH. The slight decrease in height after 4 hrs may be due to further packing of the loosely sedimented flocs. Minimum height for pH = 10 is an indication of optimum dispersion and ideal packing whereas intermediate heights for pH values of 7 and 12 are due to partial agglomeration of particles. The continued increase in heights for pH values 7, 10 and 12 show that the finer particles remain under suspension for long times. This is supported by the fact that at these pH values the suspensions remain turbid even after a week, whereas the suspensions become clear within 1–2 days for the pH range of 2–5. This is brought out more clearly in Fig. 4 by measuring the total height of the slip as the zero - time height and subsequently considering the maximum level of turbidity (solids sedimented plus the solid particles held in suspension below the clear liquid) as the sediment heights at different time intervals. As one approaches the pH of flocculation (pH = 2–5) the slip is stable for hardly a day, whereas towards the pH of optimum dispersion (pH = 10) the height of suspension remains unchanged for several days.

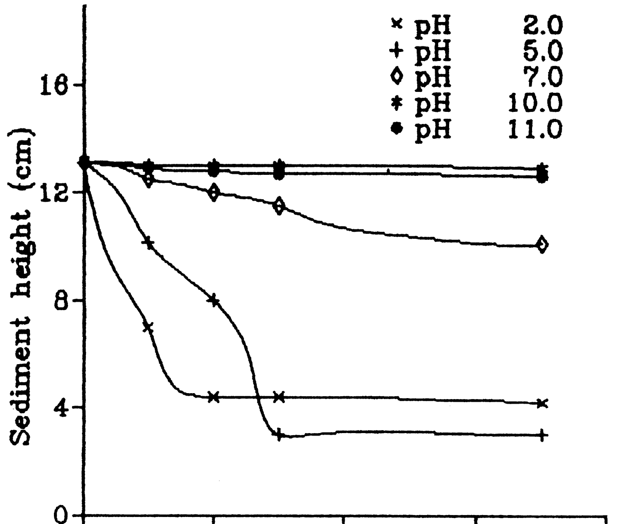

Fig. 4. Sedimentation height as a function of time for silicon carbide (G1200/2) at various pH values plotted after Adair et al. [21].

3.3. Rheological behaviour

The viscosity of 52 wt% SiC slips measured as a function of pH at a shear rate of 2702 s^{-1} is presented in Fig. 5. Lower viscosities are observed in the pH range 7–11 with a minimum at pH value of 10 for all powders. This shows that the powders are fairly well dispersed in the pH range 7–11, with the most optimal dispersion observed at pH = 10. The higher viscosities in the lower pH range (pH = 2–6) as well as at the higher pH end (pH = 11–12) is the characteristic of a flocculated suspension. Lower viscosities observed for SiC G800/2 powder throughout the pH range can be attributed to the wider particle size distribution of these powders with a fair amount (≈ 15%) of submicron particles present in them. That even very small amount of finer particles added to coarse particles in a suspension drastically

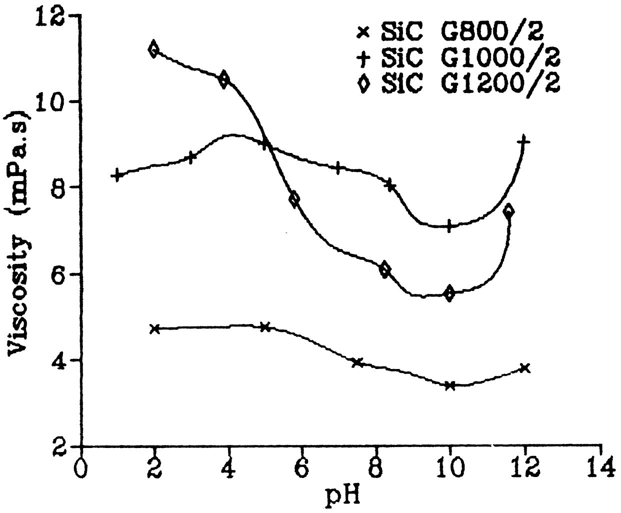

Fig. 5. Viscosity of silicon carbide slips as a function of pH.

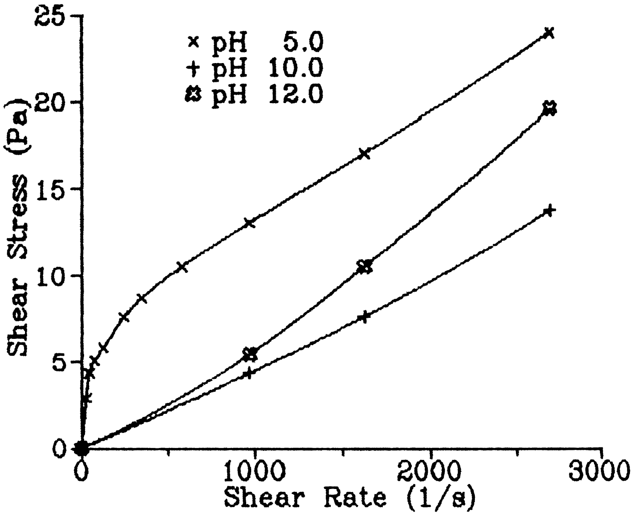

Fig. 6. Shear rate versus shear stress flow curves for silicon carbide (G1200/2) slurry as a function of pH.

decrease the viscosity is a behaviour also reported by several other authors [8,23–25].

The rheological behaviour of SiC G1200 slips at 52 wt% solid loading was further studied by measuring the viscosity and shear stress at varying shear rates and at different pH values of the slip (Figs. 6 and 7). At a pH of 10, the slip is relatively newtonian (linear shear stress versus. Shear rate curve passing through the origin, see Fig. 6) and its viscosity is constant at all shear rates (Fig. 7), indicating a well dispersed, homogeneous slip. The flow curves at pH = 5 show the characteristics of a highly flocculated suspension with a yield stress of about 7Pa and display a shear thinning behaviour. This is because, liquid is immobilised in the interparticulate pore spaces of the flocs and floc networks of the suspension, resulting in an increased effective solid loading relative to a well dispersed suspension. On application of a shear stress, shear thinning flow results due to the breakdown of the flocculated structure and the release of entrapped liquid [24]. At a higher pH value of 12, a slight shear thickening behaviour (dilatant flow) characteristic of heavy solid loaded suspensions or suspensions in which very large inter-particle electrostatic repulsive forces exist, is observed [18,24].

The effect of solid loadings on the flow behaviour of SiC G1200 slurry in deionised water at pH = 10 is presented in Fig. 8. Almost near Newtonian behaviour was exhibited at lower solid loading of 52 wt% whereas shear thickening behaviour with thixotropy was observed at solid loadings of 57 and 62 wt%. The dilatancy of highly solid loaded suspensions could be attributed to either shear induced flocculation or to a transition from ordered to disordered flow. This effect is more pronounced and occurs at lower shear rates at higher solid loadings since the structural changes require less shear energy due to smaller interparticulate separation on an average [24,25].

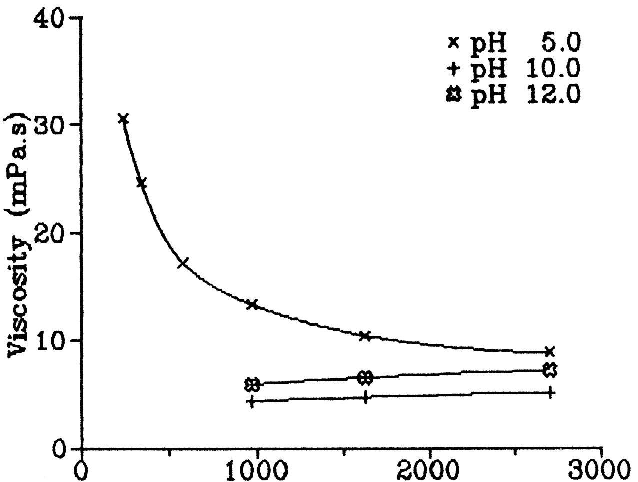

Fig. 7. Viscosity versus shear rate flow curves for silicon carbide (G1200/2) slurry as a function of pH.

3.4. Zeta potential measurements

Zeta potential, the potential in the shear plane between the charged surface of the particle and the electrolyte, reflects the interface characteristics and expresses the potential due to the net effective charge on the surface of particles in a suspension. With other factors constant, higher zeta potential is a measure of improved dispersion resulting from increased interparticular electrostatic repulsion between the like surface charges acquired by the particles in the medium [13,15].

The zeta potential measured for three SiC powders presented in Fig. 9 shows almost similar behaviour with negative zeta potential values over a substantial pH range demonstrating the acidic character of SiC. All the three powders show a maximum in the zeta potential values at PH = 10 indicating that the slip is well dispersed at this pH. The three powders G800, G1000 and

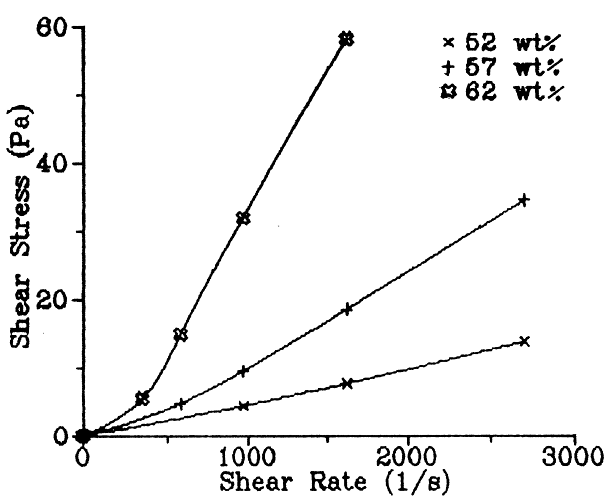

Fig. 8. Shear rate versus shear stress curves for silicon carbide (G1200/2) slurry at pH = 10 as a function of solid loading.

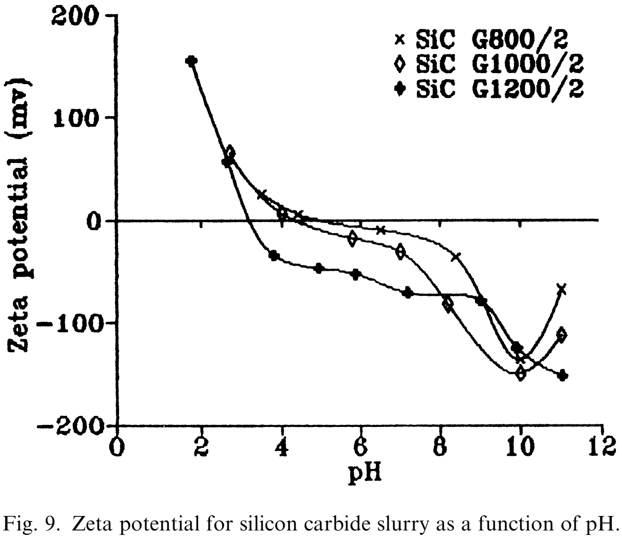

Fig. 9. Zeta potential for silicon carbide slurry as a function of pH.

G1200 have their isoelectric points (IEP) (i.e. the pH at which the net charge on the particle surface is zero) at the pH values of 4.8, 4.2 and 3.3 respectively. These results are in agreement with those already reported [14,16–21]. The surface of SiC particles are usually covered by a thin film of silica with acidic silanol sites on its surface and depending on the percentage of oxide layer present the isoelectric points of SiC powders approach that of silica. Silica itself is known to posses an IEP in the pH range of 2–3.7. The lowest IEP of 3.3 for G1200 powder is attributable to the highest percentage of surface oxidation (hence to surface silica) owing to the finer particle size distribution of these powders. At the isoelectric points (IEP), due to the absence of repulsive forces, the slip is highly flocculated and exhibits non-Newtonian flow behaviour, higher viscosity, faster sedimentation rate and a larger sedimentation height/volume. At the high pH value of 11, a substantially high ionic strength increases the charge in the liquid phase and reduces the potential difference between the powder surface and liquid medium, resulting in a decrease of the absolute value of zeta potential. As a manifestation of this effect the SiC slips at pH > 10 show a small degree of flocculation with a slight non-Newtonian behaviour (Figs. 6 and 7), coupled with larger sedimentation heights (Figs. 2 and 3).

Table 2
The casting conditions and properties of slip cast green bodies

pH	Solid loading (wt%)	Releasing time (min)	Moisture content (%)	Green density	
				(gm/cm^3)	(%)[a]
4	50	120	24.3	1.568	49.0
6	50	120	20.8	1.573	49.2
8	50	30	14.9	1.809	56.5
	58	50	20.0	1.627	50.8
9	50	40	12.7	1.949	60.9
	58	40	12.0	1.944	60.8
10	50	45	12.3	2.009	62.8
	58	30	12.9	1.977	61.8
	64	30	12.7	1.963	61.3
11	50	30	16.5	1.913	59.8

[a] Percentage theoretical density.

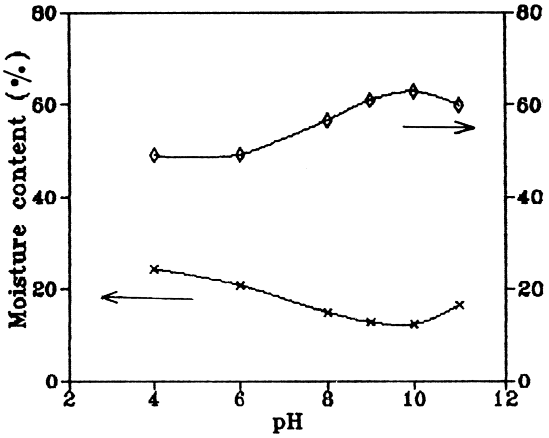

Fig. 10. Percentage moisture content and density (%theoretical) of slip cast green compacts of silicon carbide (G1200/2) as a function of pH.

3.5. Slip casting

Under specific conditions of slip casting, the release time, the density and the moisture content of slip cast

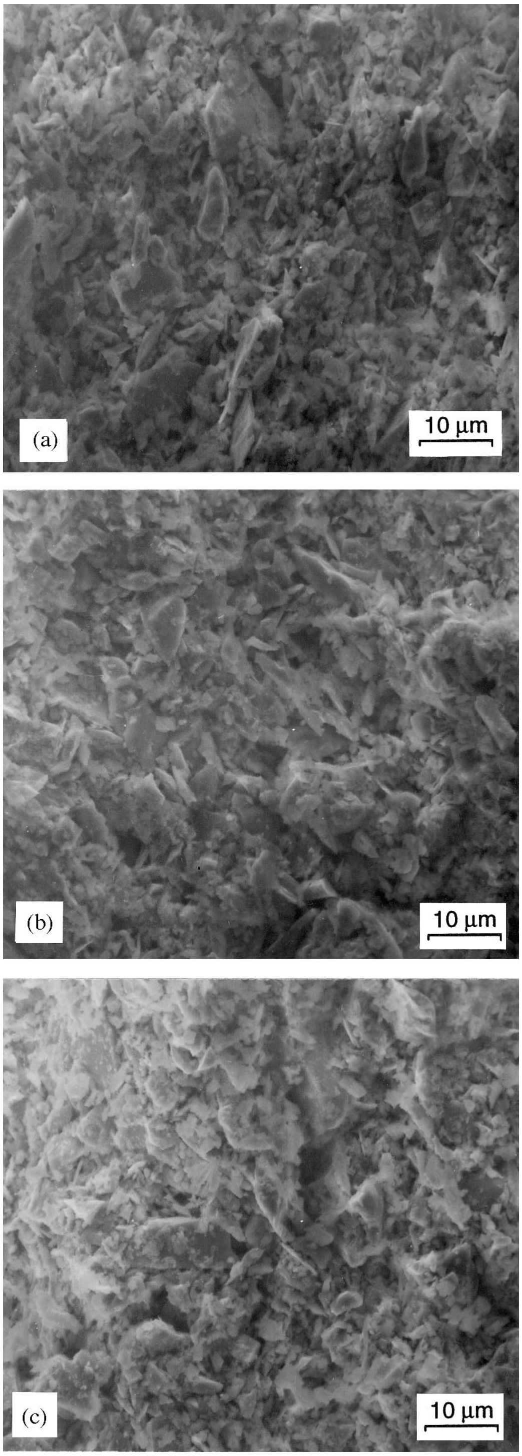

Fig. 11. SEM photomicrographs of fractured surfaces of slip cast SiC (G1200/2) green body: (a) pH = 4, (b) pH = 8, (c) pH = 10.

green bodies are presented in Table 2 and Fig. 10. These data correlate very well with the results obtained through particle size analysis, sedimentation, viscosity and zeta potential measurements. The slips could take higher solid loading in the pH range of 7–11, a region of moderate to fairly good dispersion, whereas, in the acidic pH range only lower solid loadings were possible due to considerable flocculation effects. Highest moisture content of 24% and lowest density of 49% (of theoretical) were observed at the pH of 4 (near IEP) whereas the lowest moisture content of 12% and highest density of 63% where observed at the pH value of 10 at which the dispersion (deflocculation) was maximum. The scanning electron micrographs of fractured surfaces of green compacts obtained from flocculated (pH = 4 and 8) and from well dispersed (pH = 10) slips presented in Fig. 11 (a-c) reveal higher percentage of porosity and increased agglomeration due to nonhomogeneous packing at the pH values of 4 and 8 whereas a more uniform dense packing without agglomeration at the pH value of 10. These results explain the pH controlled dispersion quality of SiC powders in aqueous media. The addition of dispersion aids are known to profoundly influence the dispersion quality of the SiC slips and therefore would superpose their influence on this simple pH dependent dispersion behaviour. Our studies would now be continued to evaluate the influence of the dispersing aids on the dispersability of SiC slips.

4. Conclusion

It is revealed by the present studies that the degree of dispersion and hence the stability of SiC slips in an aqueous medium could be controlled by merely adjusting the pH of the medium. The dispersion of commercial silicon carbide powders in deionised water was studied in the pH range of 2–12. The slips showed moderate to fairly good dispersion in the pH range 7–10. The highest dispersion/deflocculation was observed at the pH value of 10, as revealed by a minimum in viscosity and sedimentation height and by a maximum in zeta potential for these slips. A high degree of flocculation was observed in the acidic pH range of 2–6 (particularly on either side of the isoelectric points) as revealed by near-zero zeta potential values. At these pH values the slips show a shear thinning behaviour, faster sedimentation rates and higher sedimentation heights. Suspensions with good dispersion (strong interparticulate repulsive forces) at pH = 10 and 11 show near-newtonian behaviour up to intermediate solid loadings of 50 wt%, whereas at higher solid loadings they become shear thickening. Highly solid loaded slips at pH = 10 result in green bodies with uniform dense packing of SiC particles with highest green density and lowest occluded moisture content showing it as the condition of optimum dispersion. On the contrary, at lower pH values (viz. 2–6) which constitute the condition of flocculation, highly agglomerated green compacts with high moisture content and much lower green densities resulted. These results demonstrate that the colloidal processing through control of pH of SiC suspension could be used as a recommended route for obtaining high solid loaded, well dispersed slips leading to uniform and dense slip cast green bodies.

Acknowledgements

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