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Changes in β-sialon suspension behaviour due to surface oxidation, milling, surfactant and sintering additive

A. Kudyba-Jansen [a,b], M. Muurmans [a], M. Almeida [c], J. Laven [d], H.T. Hintzen [a,*], R. Metselaar [a]

[a]Laboratory of Solid State and Materials Chemistry, Eindhoven University of Technology, PO Box 513, 5600 MB Eindhoven, The Netherlands
[b]Material Science Department, Silesian Technical University, Katowice, Krasinskiego 8B, 40-019 Katowice, Poland
[c]Department of Ceramics and Glass Engineering, UIMC, University of Aveiro, 3810 Aveiro, Portugal
[d]Laboratory of Coatings Technology, Eindhoven University of Technology, PO Box 513, 5600 MB Eindhoven, The Netherlands

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Abstract

Results of a study of the behaviour of aqueous suspensions of carbothermally prepared β-sialon are presented. The influence of an oxidation treatment, attrition milling, sintering additive (Y_2O_3) and addition of a deflocculant (Dolapix CE 64) on the stabilisation behaviour of the suspensions was studied. Obtained results show that the surface properties of β-sialon particles are affected, as expected, by the oxidation treatment (isoelectric point, $pH_{iep} \approx 2.1$) and by attrition milling of the power ($pH_{iep} \approx 4.6$). In the presence of Y_2O_3 the suspension of β-sialon becomes less stable ($pH_{iep} \approx 3.4$), the viscosity is increased and the stable pH range is reduced. This behaviour is explained in terms of surface composition of β-sialon, the solubility of yttria in acidic solution and the possibility of precipitation of yttrium-hydroxy complexes on β-sialon surface at higher pH while increasing the pH. Addition of the deflocculant increases the absolute zeta potential and changes the suspension behaviour from shear thickening to shear thinning. Low viscosity suspensions with 60 wt% β-sialon and 10 wt% Y_2O_3 can be prepared at $pH > 10$, such that they can be used for slip casting.

Keywords: A. Suspensions; D. Sialon; D. Y_2O_3; Rheology; Zeta potential; Stability

1. Introduction

Rapidly developing technology requires the introduction of new materials as well as improvement on existing ones. One of the best known high performance engineering materials is the Si_3N_4 ceramic that has very good mechanical properties at high temperatures. Simultaneous equivalent substitution of Si–N by Al–O in the Si_3N_4 lattice resulted in a new material called sialon, with general formula $Si_{6-z}Al_zO_zN_{8-z}$ [1]. The material has several advantages over Si_3N_4 like easier densification, better chemical and oxidation resistance.

Engineering ceramics are often expected to be fully densified, highly reliable and preferably low-cost, even for complex shapes. Expected final ceramics properties will only be attained by controlling the microstructure of both green and final products, which are strongly related to powder characteristics, compaction processes and sintering conditions.

Slip casting has been reported as a suitable, inexpensive consolidation process to produce materials with high green densities and homogeneity of microstructure, even for complex geometries [2–8]. A good casting slip should meet a number of commonly known requirements [9], viz. it must be pourable, have high solid concentration and the particles must not settle out, meaning the deflocculation degree has to be controlled. Therefore, obtaining a stable, sufficiently dispersed, uniform and highly concentrated slip of a powder in water is a particularly critical step [8,10]. Slips with the required properties will be achieved only if the surface chemistry of the powder is known and understood [11,12]. In this respect sialon powders in particular those with high *z*-values, may differ considerably from silicon nitride. In the present paper we discuss the behaviour of carbothermally prepared sialon with $z = 3$. For all carbothermally produced materials a heat treatment in air has to be applied in order to

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* Corresponding author. Tel.: +31-40-247-3113; fax: +31-40-244-5619.
E-mail address: h.t.hintzen@tue.nl (H.T. Hintzen).

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remove residual carbon. This again influences the surface properties and leads to differences of suspension behaviour in comparison with commercially available powders.

In order to understand and manipulate the behaviour of slips and surface properties, zeta potential and rheometry measurements can be applied. The colloidal stability of a suspension is directly related to the magnitude of the zeta potential of the particles. High absolute values of the zeta potential improve the degree of deflocculation of the suspension. At the same time, an increase of the volume fraction of the agglomerates results in an increase in its viscosity. Adjusted dispersion degree is expected to result in an optimal particle packing, high density and homogeneous microstructure of the green bodies, necessary for obtaining high quality sintered products [10].

In our previous work [13,14], we compared the suspension (prepared by attrition milling) behaviour of Ca-α-sialon and β-sialon powder (with and without surfactant) as a function of pH. After addition of deflocculant both suspensions behaved similarly. They had pH_{iep} around 5 and the highest absolute zeta potential values and the lowest viscosities were found at pH 10–11.

Here we present an extended study on the influence of a heat treatment in air and of attrition milling on the suspension behaviour. Y_2O_3 is a well known sintering additive for sialon powders. Since addition of yttria may influence the suspension behaviour, we also performed rheological and zeta potential measurements with and without yttria.

2. Methods

2.1. Powder and suspension preparation

β-sialon powder with approximate composition $Si_3Al_3O_3N_5$ used in this study was prepared carbothermally starting from clay and hammer crushed afterwards. The preparation has been described elsewhere [13]. The powder was heated up to 750°C in static air with a heating rate of 10°C/min and kept at this temperature for 0–25 h in order to remove any residual carbon.

The standard way of the suspension preparation was as follows: after heat treatment for 10 h (carbon removal) β-sialon (200 g) was attrition milled (HD 01, Union Process) at a speed of 300 rpm for 6 h in ultra pure water (133 g) (Fistreem Cyclon) using a 500 ml Teflon coated tank and 750 g Si_3N_4 milling balls (5 mm diameter). The resulting suspensions were sieved over a screen with openings of 0.315 mm diameter. After sieving, the suspension had an initial solid content of about 60 wt%. The pH of the β-sialon suspension after attrition milling is about 9. Suspensions with lower solid contents were obtained by dilution with water to the required percentage and subsequent ageing for 24 h on a roller bench. Unless stated otherwise, pH of individual suspensions for zeta potential or viscosity measurements were adjusted with HNO_3 and NH_4OH solutions starting from the initial pH (≈ 9).

In this study yttrium oxide (HC Starck) of 99.95% purity and median grain size $d_{50} = 0.8$ μm was used as a sintering additive and ammonium polyacrylate, Dolapix CE64 (Zschimmer & Schwartz) as a surfactant. Various amounts of either additive were added into small bottles containing the attrition milled β-sialon powder (pH≈9) and subsequently left on a roller bench for 24 h.

The following β-sialon suspensions were investigated as a function of pH:

- without and with sintering additive Y_2O_3 (1–10 wt% relative to β-sialon content)
- without and with heat treatment at 750°C in air (0–25 h) but no attrition milling was applied
- without (suspended by rolling) and with attrition milling of the 10-h oxidised powder
- without and with surfactant (0–2 wt% relative to β-sialon content)
- with different solid loading 40, 50 or 60 wt% β-sialon (10 wt% Y_2O_3 and 0.6 wt% surfactant)

2.2. Investigation techniques

The particle size, particle size distribution and zeta potential (ζ) as a function of pH (3–11) were measured. A Zeta Sizer 4 (Model AZ 6004, Malvern Instruments, UK) based on quasi elastic light scattering (ELS) was used. Measurements were performed employing diluted suspensions (≈1 wt%) which were obtained by adding a small drop of a concentrated (60 wt%) suspension to a 0.001 M NaCl solution. This solution was used to increase the stability of the velocity profile of particles in the applied electric field, and to maintain a constant ionic strength, with a consequent constant double layer thickness.

The rheological measurements were performed using a Carrimed CSL Rheometer (Carrimed House, UK), fitted with a cone and plate geometry. The measurements were performed at 20°C in a shear sweep mode at shear rates ($\dot{\gamma}$) of 1–1000 s^{-1}. The viscosity ($\eta = \tau / \dot{\gamma}$) at 1000 s^{-1} and yield stress (τ_y) of suspensions as a function of pH (3–10) were analysed using the Herschell–Buckley model [15]. This model is described by the following equation:

$$\tau = \tau_y + K_p \cdot \dot{\gamma}^n$$

where: τ the stress, τ_y the yield stress, K_p the plastic viscosity coefficient, $\dot{\gamma}$ the shear rate and n the shear rate index.

3. Results and discussion

3.1. The influence of heat treatment

The carbothermally prepared β-sialon powder needs to be heat treated in air in order to remove residual carbon. The influence of this treatment on the surface properties of β-sialon was investigated by zeta potential measurements. Fig. 1 shows the zeta potential values of β-sialon powders that were heat-treated in air at 750°C for different times and dispersed in water without attrition milling. Heat treatments between 0 and 10 h cause a shift of the isoelectric point of β-sialon to lower pH. A prolonged time, up to 25 h, did not change any further the zeta potential values (not incorporated in Fig. 1). This indicates that the 10-h heat treatment is sufficient to reach a constant composition of the β-sialon surface. From an XPS and SIMS study we found that the heat treatment of β-sialon in air significantly oxidises the surface, and oxygen rich compounds (aluminium silicates) are formed [16]. After a long oxidation treatment segregation of silicon on the outermost surface can occur which is most probably present in the form of silica, what is in agreement with the results of Lee et al. [17]. This was reflected in the pH_{iep} value of 2.1 which is close to the one of SiO_2 (2.0) [21].

At high pH values the oxidised surface of β-sialon has a higher absolute value of the zeta potential as compared to the non-oxidised one, which is very profitable for obtaining well-dispersed powders. The shift of the pH_{iep} to the acid side is due to an increase of the silanol/silizane ratio on the surface [22].

3.2. The influence of attrition milling

The zeta potentials of dry milled and oxidised powders, versus those of dry milled, oxidised and attrition milled in water are presented in Fig. 2. It shows a shift of pH_{iep}, from 2.1 to 4.5, due to attrition milling of β-sialon. The absolute value of the zeta potential of powder without attrition milling in water is higher than the one with attrition milling at high pH. This implies a significant difference in the surface characteristics of β-sialon in water after attrition milling. During attrition milling in water fresh surfaces are created due to fracture. These surfaces are nitrogen rich and thus NH and NH_2 are present at the surface. The attrition milled powder in water is covered by more basic amino groups resulting in a higher pH_{iep}, while the surface oxidisation of the powder leads to coverage by more acidic -OH groups, i.e. a more silica like surface ($pH_{iep}=2.0$). The presence of amino groups is undesirable since these groups form nitrogen-bridged hard agglomerates, while silanol groups form hydrogen-bridged soft agglomerates

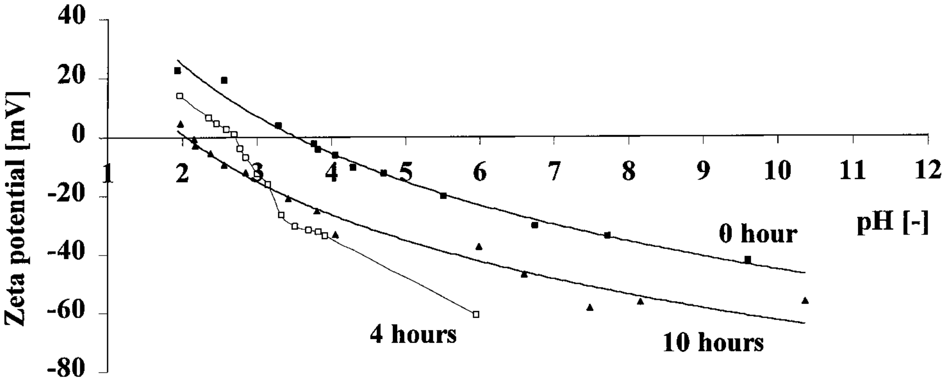

Fig. 1. Effect of heat treatment on the zeta potential of suspension of β-sialon (surfactant free) heat treated at 750°C for 0, 4 and 10 h (without attrition milling).

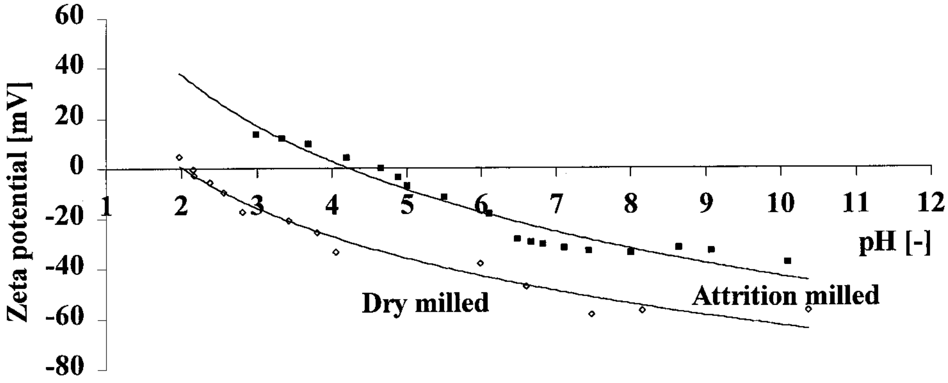

Fig. 2. Zeta potential of β-sialon suspension (surfactant and Y_2O_3 free) dry hammer crushed and heat treated and dry hammer crushed, heat treated and attrition milled.

[18]. Also the adsorption of ammonium polyacrylate surfactant is hindered [19]. Unfavourably, due to the attrition milling of the powder the positive effect of oxidation is lost due to generation of hard agglomerates and higher pH_{iep}. As the hard agglomerates can increase both inhomogeneity and porosity of the green product, their occurrence has to be prevented by, for example, addition of surfactant although its effectivness can be held up. Alternatively, a dry milling procedure can be applied instead of the wet attrition milling, or the sialon's surface can be intentionally covered with Y_2O_3 or SiO_2 after the milling.

3.3. The influence of Y_2O_3 as a sintering additive

The zeta potentials of pure β-sialon (10-h heat treatment and attrition milled) suspensions and of Y_2O_3 (as received) suspensions showed that these powders are oppositely charged between pH 4.6 and 9.0 (Fig. 3). In this pH range, the powders can heterocoagulate. Below pH 4.6 and above pH 9.0, the particles in suspension have the same charge so the heterocoagulation is not expected.

In a suspension containing β-sialon and Y_2O_3 the way pH is adjusted plays a significant role. With decreasing pH the zeta potential gradually increases (Fig. 4), which is similar to the behaviour of β-sialon without Y_2O_3. However, for increasing pH, the curve looks completely different. After an initial decrease of the zeta potential, at about pH 4.5 it starts to increase again, which indicates a change in surface characteristics. Obviously, the Y^{3+} ions, originating from the dissolution of Y_2O_3 [20] at low pH, start to precipitate due to surpassing the solubility limit of $Y(OH)_3$. This process will preferentially take place at the surface of the sialon grains as a consequence of hetergenous nucleation. During further increase of the pH, the zeta potential remains increasing up till the point where the precipitation has been completed (pH 5.5: Fig. 4). Subsequently the zeta potential again shows a downwards trend for higher pH values, characteristic for the $Y(OH)_3$ covering partially the sialon grains. At high pH the sialon surface is covered with hydroxide and the measured zeta potential can be attributed to the mixed substrate and precipitate. This interpretation is supported by similar observation

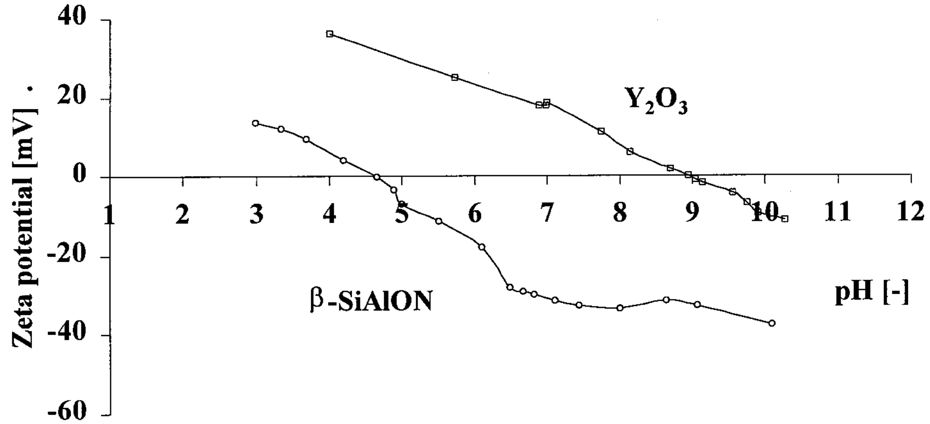

Fig. 3. Zeta potential of β-sialon (10 h heat treatment, attrition milled) and Y_2O_3 suspension as a function of pH (no surfactant).

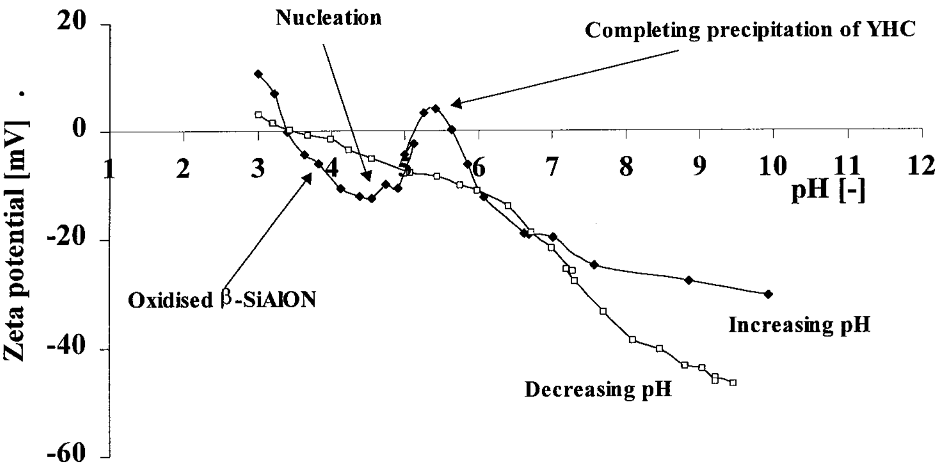

Fig. 4. Zeta potential of β-sialon suspension (10 h heat treatment, surfactant free) with 10 wt% of Y_2O_3, measured with either increasing or decreasing pH.

reported in the literature. Similar local maximum was described by James and Healy [21] for the case of precipitation of hydrolyzable metal ions at oxide–water interfaces and by Hackley and Malghan [22] for Si_3N_4 with $Y(NO_3)_3$. It was observed that an yttrium–hydroxy complex (YHC) is formed on Si_3N_4 substrate during acid-base titration of Y^{3+} nitrate solution. Precipitation of YHC depends on metal ion concentration, pH, ionic strength and particle surface area [22].

The above mentioned was studied for β-sialon/Y_2O_3 suspensions with a varying amount of Y_2O_3 (Fig. 5). All curves in Fig. 4 show the occurrence of a local maximum in the zeta potential between pH 5 and 6. The isoelectric points of β-sialon with different Y_2O_3 contents are presented in Table 1.

In alkaline solutions, the precipitation is not expected. However, some interaction with the negative β-sialon could occur due to presence of minor species ($Y_2(OH)_2^{4+}$, $Y(OH)^{2+}$ and Y^{3+}) which are in equilibrium with the hydrolysed Y_2O_3 to $Y(OH)_3$ [23–25].

The influence of Y_2O_3 on the rheological properties of the suspensions was also investigated. The variation of the viscosity ($\gamma = 1000\ s^{-1}$) with the pH of the suspending medium for the β-sialon slurries (attrition milled, no deflocculant) with different additions of yttria is shown in Fig. 6. For all curves the maximum in viscosities is observed between pH 5.5 and 8.5 that is between the pH_{iep} of β-sialon and that of Y_2O_3 where heterocoagulation takes place. With increasing amount of yttria, both the absolute value of the viscosity and the pH of the viscosity maximum increase (Table 1). At the same time the yield stress increases. As shown in Fig. 6 the pH range, where the suspension has the highest viscosities, increases with the increase of the Y_2O_3 content.

In our previous investigation [14] the viscosity maximum corresponded to the pH_{iep} determined by zeta potential measurements. Here the relationship is not straightforward. The observed shifts in the pH where a maximum viscosity occurs, clearly show that the particle interaction changes with increased yttria content. We did not study this effect in more detail and it is therefore difficult to say which interaction plays a dominant role (van der Waals, attraction, electrostatic electrical double layer or solvation forces) [26].

From Fig. 6 it can also be seen that the pH range where the suspension is unstable increases with the increase of the Y_2O_3 content and Fig. 7 shows the pH range, where it is possible to obtain stable suspensions. This range is influenced by the Y_2O_3 content. Here a stable suspension was defined as possessing a viscosity

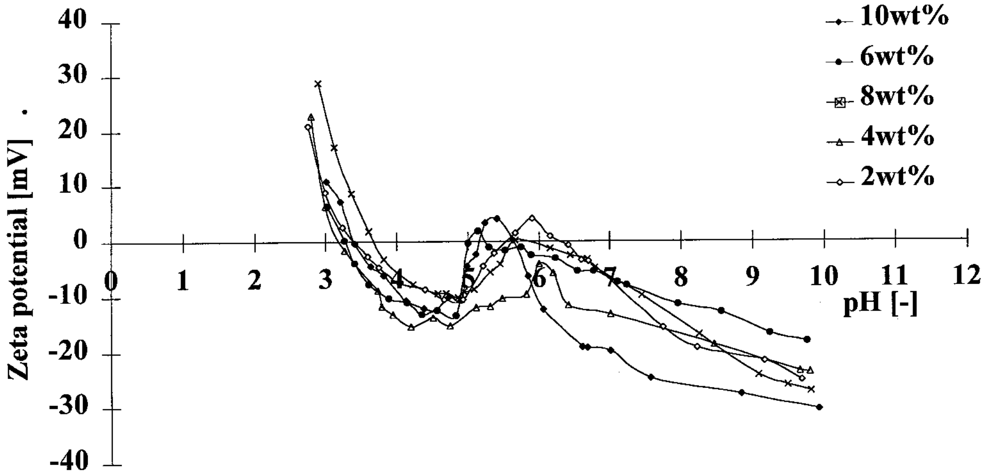

Fig. 5. Zeta potential of β-sialon suspensions (10 h heat treatment, surfactant free) with 2, 4, 6, 8 and 10 wt% of Y_2O_3 measured with increasing pH.

Table 1
Characteristics of 60 wt% β-sialon suspension (10 h heat treatment, attrition milled and surfactant free) with different amounts of Y_2O_3

Y_2O_3 (wt%)	pH_{iep} from zeta potential (pH)	Maximum viscosity (pH)	Maximum viscosity value (Pa s)	Maximum yield stress (Pa)
Pure Y_2O_3	9.0	–	–	–
0	4.6	5.4	0.04	9
2	3.4	5.8	0.06	11
4	3.3	≈6.0	0.08	16
6	3.3	≈6.5	0.10	17
8	3.6	≈6.5	0.16	21
10	3.4	≈6.5	0.14	19

($1000\ s^{-1}$) below 0.02 Pas and a low yield stress, below 2 Pa. This condition corresponds to suspensions with a low degree of flocculation. The stability pH ranges are dependent on the Y_2O_3 content, as already concluded from Fig. 6 and are in agreement with the results obtained for zeta potential measurements (Fig. 4). The lower pH range is unsuitable due to dissolution of yttria [27].

From all the results a concentration of 10 wt% was selected to be used in the preparation of the suspensions.

3.4. The influence of surfactant

In order to stabilise and prevent ageing of the suspension and prevent eventually inhomogeneous green product, a surfactant can be added. The influence of the Dolapix CE64 as a surfactant was investigated. The zeta potential measurements on β-sialon attrition milled with and without deflocculant are shown in Fig. 8. The pH_{iep} did not change significantly but the absolute zeta potential values increased, with a more pronounced effect for pH values higher than 8. Similar effect of the surfactant on zeta potential of the diluted suspension containing 60 wt% β-sialon with 10 wt% Y_2O_3 is shown in Fig. 9. The crossing point was shifted from pH 5.7 to 6.8 with the addition of Dolapix CE64 (Fig. 9) and the absolute values above pH 8 were increased implying better deflocculation.

From the viscosity and yield stress measurements (Figs. 10 and 11) it can be seen that 0.2–0.6 wt% of surfactant is requested to deflocculate a 60 wt% β-sialon suspension with 10 wt% Y_2O_3 at pH 10.6 (Fig. 10). Further addition of Dolapix CE64 caused a slight increase in viscosity (probably due to secondary and tertiary electroviscous effects). Within this range of surfactant levels, the corresponding shear rate indices were about 0.8 in all cases except for the suspension without Dolapix, for which a value of 1.25 was found. Apparently Dolapix effectively changes the rheological behaviour of the suspension from shear thickening to shear thinning, which is favourable for slip casting. Those results allow us to

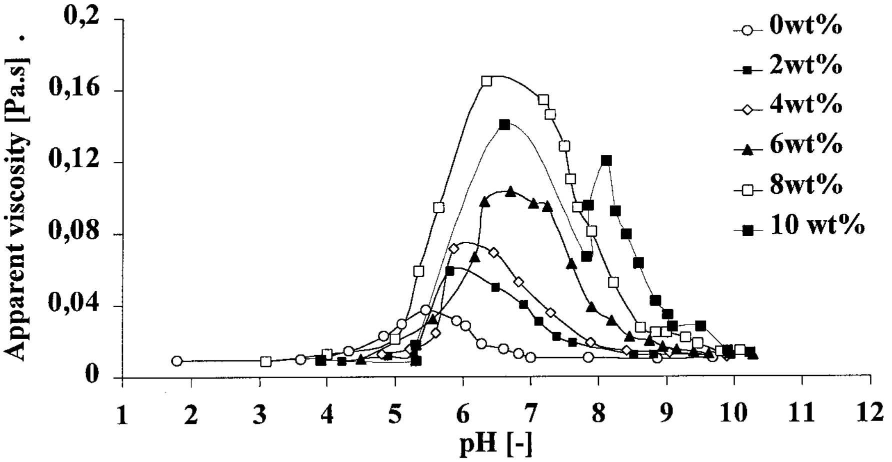

Fig. 6. Viscosities of β-sialon suspension (10 h heat treatment, surfactant free) with different amounts of Y_2O_3 (0–10 wt%) measured at shear rate $1000\ s^{-1}$ as a function of pH.

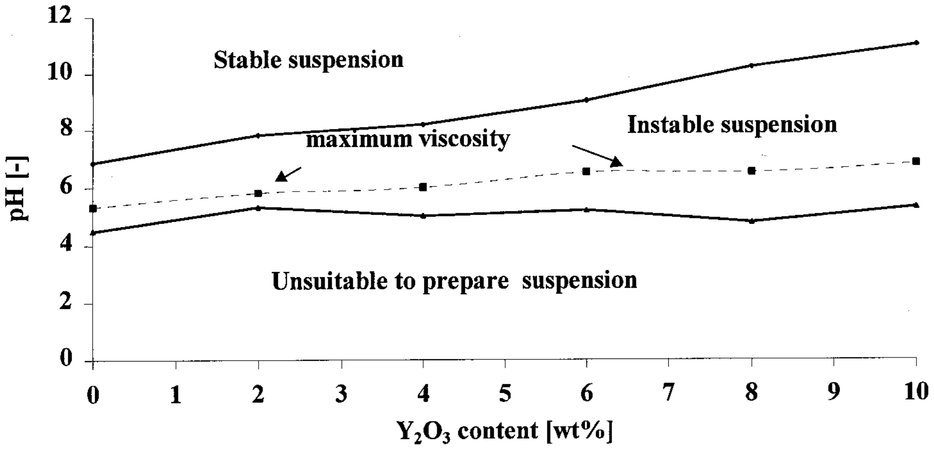

Fig. 7. Stability regions of β-sialon suspensions (surfactant free) with different Y_2O_3 additions and showing the pH of the maximum viscosity.

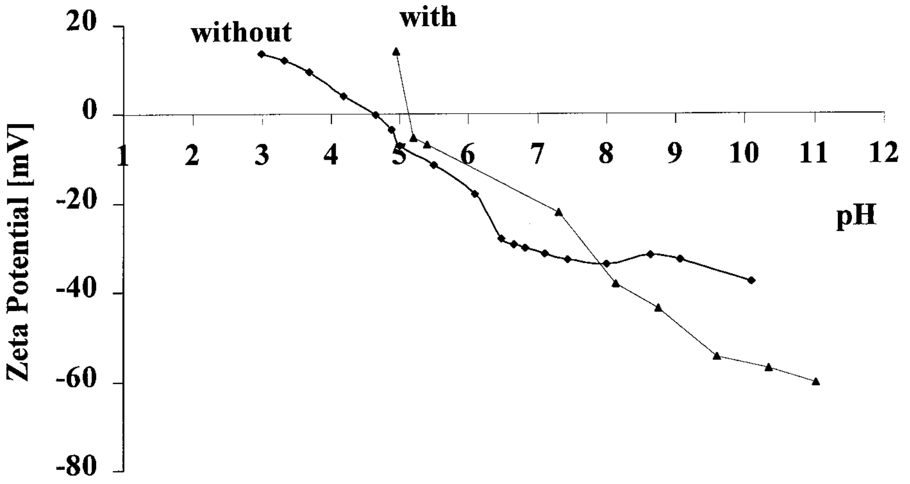

Fig. 8. Zeta potential of β-sialon (10 h heat treatment and attrition milled) with and without Dolapix.

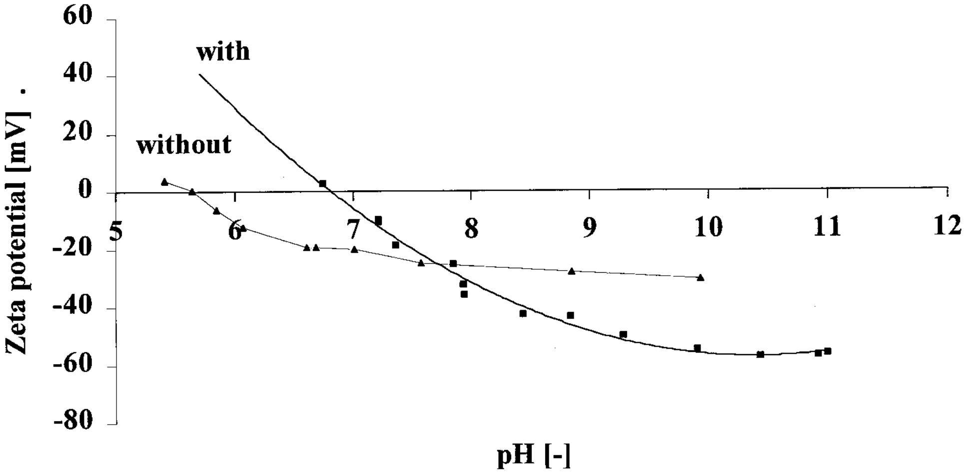

Fig. 9. Effect of surfactant (Dolapix CE64) on the zeta potential of a 60 wt% β-sialon suspension with 10 wt% Y_2O_3 (diluted to the necessary concentration with supernatant) measured with increasing the pH.

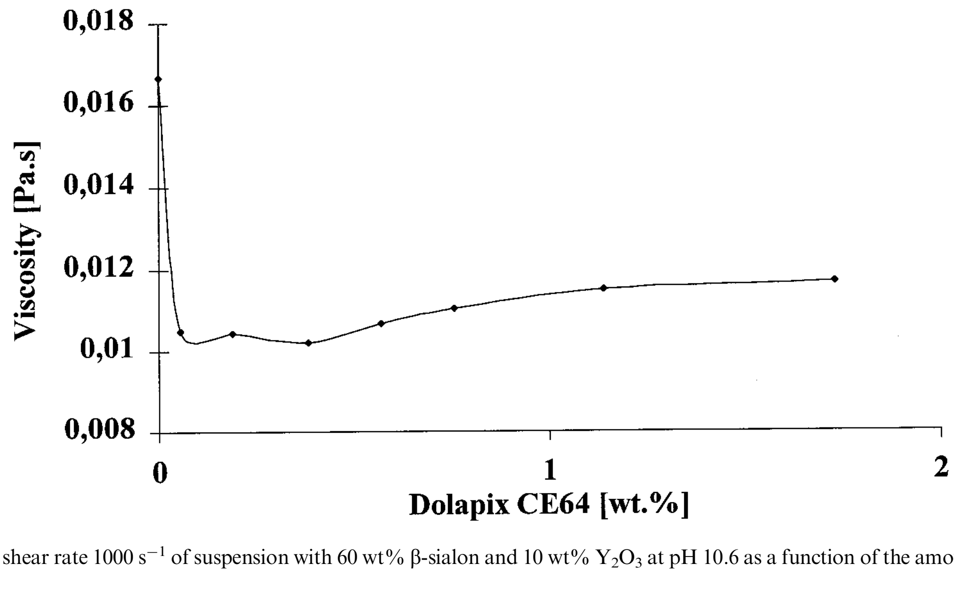

Fig. 10. Viscosity at shear rate 1000 s^{-1} of suspension with 60 wt% β-sialon and 10 wt% Y_2O_3 at pH 10.6 as a function of the amount of Dolapix CE64.

conclude that the addition of Dolapix to the suspensions results in an increase in the absolute values of zeta potential in the pH range 9–11 and a stable suspension, with low viscosities and low yield stresses, can be obtained for pH 10–11.

A high solid loading suspension (60 wt%) to be used in a slip casting process should be prepared at pH 10.6 and with addition of Dolapix. Those conditions correspond to a minimum value of viscosity and a maximum zeta potential absolute value.

3.5. *The influence of solids loading*

Solid loading plays a significant role in the rheological behaviour of a suspension. The viscosity shows large variations in the maximum values for the different solid loadings (Fig. 12). β-sialon suspensions containing 50 and 60 wt% suspensions showed similar trends in their behaviour (Figs. 12 and 13). The maximum viscosity of the 40 wt% suspension is shifted to lower pH. The shift is caused by different flocculation mechanism, when the mixture of β-sialon and Y_2O_3 is diluted and the maximum is shifted towards pH_{iep} of β-sialon. The maximum flocculation was found at pH 7.4 (60 wt%), 7.9 (50 wt%) and ≈6 (40 wt%). The lowest viscosity and lowest yield stress were observed at pH 3–4 and 10–11 for the 40, 50 and 60 wt% suspensions. At the pH where the maximum viscosity is observed a maximum flocculation is expected. The maximum yield stresses observed are for increasing solid loading: 0.6, 4.8 to 16.9 Pa, respectively.

A deflocculated (i.e. stable) suspension, with low viscosity, low yield stress, the highest possible solid loading and preferably showing shear thinning behaviour is required for slip casting. Concerning the viscosity of suspensions of 60 wt% β-sialon with 10 wt% of

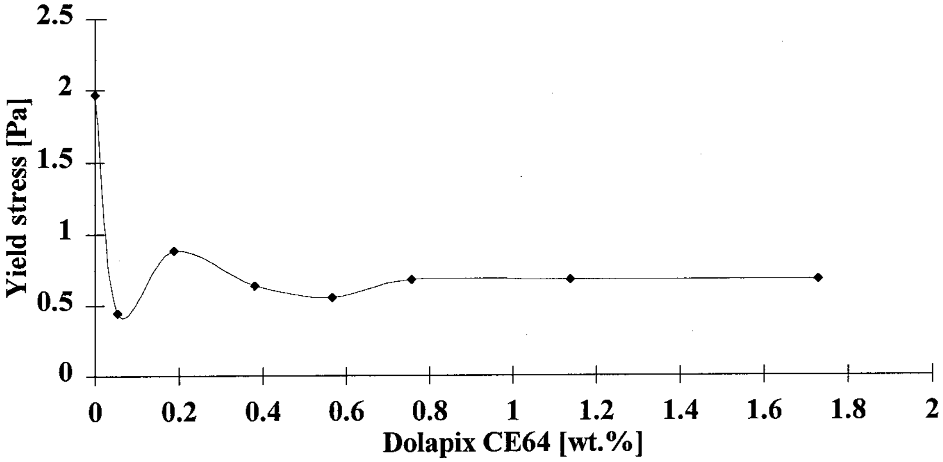

Fig. 11. Yield stress of suspension with 60 wt% β-sialon and 10 wt% Y_2O_3 at pH 10.6 measured at shear rate 1000 s^{-1} as a function of the amount of Dolapix CE64.

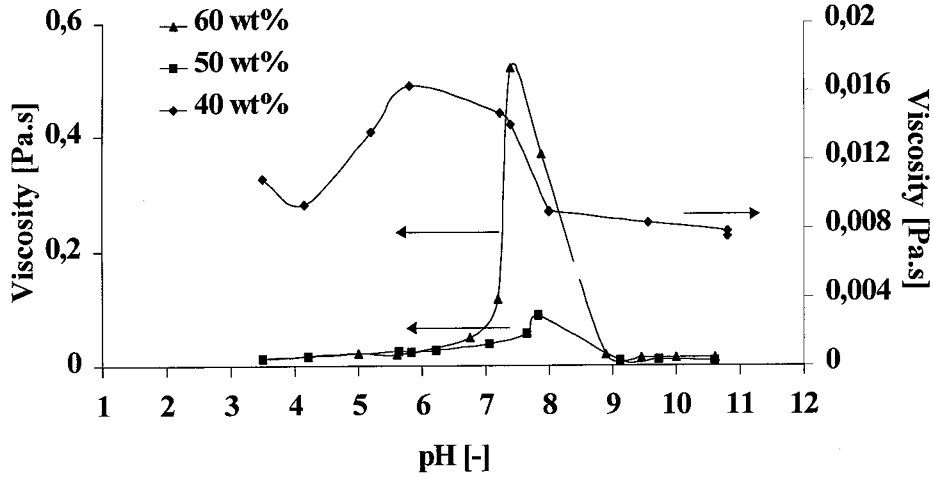

Fig. 12. Viscosity of 40, 50 and 60 wt% suspensions containing 10 wt% Y_2O_3 and 0.6 wt% Dolapix CE64 measured at 1000 s^{-1} (attrition milled).

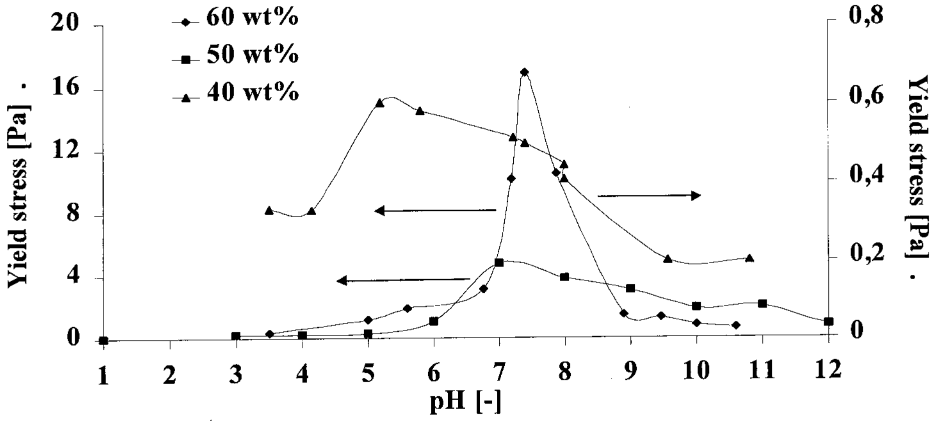

Fig. 13. The yield stress of 40, 50 and 60 wt% suspensions containing 10 wt% Y_2O_3 and 0.6 wt% Dolapix CE64.

Y_2O_3 these requirements are fulfilled at pH 3–5 and 9–11. At the basic pH the viscosity is below 0.05 Pas and the yield stress is below 1 Pa. The acidic range cannot be used due to dissolution of yttria [27], as mentioned before in Section. 3.3. Since the suspension containing 60 wt% β-sialon, 10 wt% Y_2O_3 and surfactant shows shear thinning behaviour, the low viscosity values (< 10 Pas) and possess high absolute zeta values, it can be assumed that even higher solid loading of β-sialon can be used for slip casting.

4. Conclusions

The aqueous suspension behaviour of carbothermally prepared β-sialon powder is affected by addition of sintering additive (Y_2O_3), oxidation treatment, milling and surfactant.

Pure β-sialon (heat treated and attrition milled) suspension is stable at pH 3–4 and 7–11. After addition of Y_2O_3, the rheological behaviour of β-sialon suspension is modified. If the pH is increased, the zeta potential versus pH curve becomes more complex showing a local maximum when the pH is increased. When the pH is increased the additive leads to formation of yttrium hydroxy complexes on β-sialon surface at basic pH.

Between pH 4.5 and 9 heterocoagulation occurs in the β-sialon–Y_2O_3 suspension causing high values of the viscosity and the yield stress. β-sialon with Y_2O_3 suspensions can be prepared only at pH 10–11 due to dissolution of Y_2O_3 at low pH. Addition of a deflocculant, Dolapix CE64 lowers the yield stress and viscosity values.

The heat treatment and attrition milling influence the suspension behaviour changing the pH_{iep} and zeta potential. While the heat treatment leads to an oxidised (more silica-like) surface, attrition milling has the opposite effect creating nitrogen-rich surfaces.

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