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Optimization of feedstock properties for reaction-bonded net-shape zircon ceramics by design of experiments

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Abstract

For the manufacturing of reaction-bonded ceramic microparts by low-pressure injection moulding, feedstocks with an optimized rheological behaviour are required. In order to evaluate the main influences on the rheological properties of feedstocks and their possible interactions, various compositions were systematically tested in the frame of design of experiments (DoE). For this purpose, $ZrSi_2$, ZrO_2, Al_2O_3, and MgO, and two different paraffins were used as starting materials. The influences of powder volume content, Zr/Si ratio, binder composition and processing temperature on the flowability of the feedstocks were observed in this line of experiments. A four-factorial fully fractional CCC-model was used. Finally the reliability of the computed statistical model was experimentally verified by means of two compositions, whose rheological behaviour has been predicted by the software.

Keywords: A. Injection moulding; D. Silicides; Design of experiments

1. Introduction

Finishing of sintered ceramics is a costly and time consuming effort if not even impossible in the case of microdevices. Therefore, manufacturing ceramic microparts requires particularly reliable and highly accurate shaping processes. There are several techniques described to shape and/or to replicate ceramic microcomponents [1–4]. One of these techniques is injection moulding. This technology produces green bodies of high stability and offers a high replication quality [5–8]. Especially low-pressure injection moulding (LPIM) of complex ceramic microparts is known for accurate replication with a high surface quality [9,10]. Moreover, the feedstocks can be moulded at temperatures between 60 and 120 °C due to the usage of binders with low melting points. Because of the use of silicone rubber as mould material, changes in the design can be easily implemented, and the abrasion of the mould is kept small by adequate processing parameters. For these reasons among other things the manufacturing costs for low-pressure injection moulding are comparatively low.

In addition to the shaping process, shrinkage of ceramics due to sintering can influence the accuracy and quality of devices. Higher dimensional reproducibility and accuracy can be achieved by reducing shrinkage tolerance or the total shrinkage [11]. The prospect of diminishing the sintering shrinkage is offered by reaction-bonding processes. Well known are reaction-bonded silicon nitride (RBSN) [12,13] and reaction-bonded aluminium oxide (RBAO) [14,15]. Both have in common that they derive from metal powders with a high specific surface area. Their reaction with the surrounding atmosphere during thermal processing causes an increase in volume, which reduces the sintering shrinkage. Reaction-bonded net-shape ceramics in the ternary system of Al_2O_3–SiO_2–ZrO_2 are instead based on intermetallic compounds, such as Zr_2Si, $ZrSi_2$, and $ZrAl_3$ [16,17]. They are able to compensate sintering shrinkage completely, because their increase in volume during oxidation is considerably higher than that of pure metals. The current shaping process is machining of green bodies, whose high accuracy has already been demonstrated [18,19]. However, one disadvantage of milling is the rather high loss of material it entails. An alternative shaping method is

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embossing green bodies of these ceramics [16], but embossing constrains the complexity and aspect ratio of the microparts.

As a consequence, LPIM of reaction-bonded net-shape zircon ceramics combines the advantages of a steady, flexible shaping process with the possibility of compensating the sintering shrinkage. A complete oxidation of $ZrSi_2$ is accompanied by a 65.1% increase in mass. Subsequent conversion of ZrO_2 and SiO_2 into zircon ($ZrSiO_4$) and cristobalite (SiO_2) is accompanied by an increase in volume of 116.2%. Using the equation described in [20] to evaluate the required powder volume content for achieving zero shrinkage, the calculated powder content for a ceramic solely based on $ZrSi_2$ is 46.0 vol.%. Adding an inert non-intermetallic compound such as ZrO_2 increases the required powder volume content, e.g. up to approx. 65 vol.% for ceramics with a Zr/Si ratio of 1.0. However, more additives are required for sintering at adequate temperatures and in order to vary the properties of the ceramics. By reason of the often conflicting influences on shaping and properties of the complex system, the feedstock development is intricate and effortful. However, using design of experiments (DoE) offers the opportunity to gain a maximum of information from a relatively low number of experiments.

In this paper, DoE was applied to systematically examine the usability of powder mixtures for low-pressure injection moulding of reaction-bonded net-shape zircon ceramics. The influence of binder composition, processing temperature, Zr/Si ratio and powder content on the rheological behaviour of feedstocks are investigated by purposefully varying these factors. The purpose of this experimental design is to use the findings in optimizing the rheological behaviour of feedstock in the production of net-shape reaction-bonded ceramic microparts.

2. Experimental

The main constituents of the powder mixtures are monoclinic ZrO_2 (TZ-0, Tosoh) with a specific surface area of 16.2 m^2/g, and the intermetallic compound $ZrSi_2$ (H.C. Starck). The reactive $ZrSi_2$ powder was pre-processed in an agitator bead mill to adjust the specific surface from approx. 1 m^2/g (as delivered) to >5 m^2/g. γ-Al_2O_3 and MgO powders (Merck) with high specific surfaces of 126.8 m^2/g and 25.1 m^2/g, respectively were added as sinter additives [21].

Powder compositions were calculated to obtain Zr/Si ratios of 0.64–0.96 in accordance with the guidelines of DoE. The corresponding powder mixtures listed in Table 1 were homogenized in 100 g batches in a planetary ball mill (Pulverisette 5, Fritsch) with 100 g of ZrO_2 grinding balls (10 mm diameter) in 100 g 2-propanol for 24 h. After milling, the solvent was evaporated in a rotary evaporator. Afterwards, the resulting powder mixtures were dried in a drying oven overnight in circulating air at 55 °C. A sieved powder fraction with sizes below 125 μm was used to prepare the feedstocks. The powder mixtures were characterized by physisorption (Flow Sorb II 2300, Micromeritics) measuring the specific surface areas. Evaluating the powders tap density (STAV 2003, JEL) gave an extent of their compactibility against their Zr/Si ratio.

Table 1
Compositions of starting powders of reaction-bonded ceramics.

Zr/Si ratio	Weight fraction, %			
	$ZrSi_2$	ZrO_2	Al_2O_3	MgO
0.64	76.50	18.00	5.00	0.50
0.70	70.70	23.80	5.00	0.50
0.80	62.85	31.65	5.00	0.50
0.90	56.55	37.95	5.00	0.50
0.96	53.35	41.15	5.00	0.50

The binder compositions under study were prepared with Siliplast LP65 and Siliplast LP13 (dispersant containing paraffin adapted for LPIM, Zschimmer & Schwarz, Lahnstein, Germany). Binder density was measured by a helium-pycnometer (Pycnomatic ATC, Porotec) at 20 °C. The temperature dependence of density above their melting point was measured with a Gay-Lussac pycnometer in a range between 80 and 120 °C. Calibrated pycnometers were filled with the molten wax and heated up to the measuring temperature in a heating cabinet. Excess wax was discharged through a capillary in the plug and wiped off. The mass of the cooled down pycnometer was determined and the density was calculated.

Altogether 15 different feedstock compositions were prepared according to the DoE specifications. For feedstock preparation the binder fractions of Siliplast LP13 and Siliplast LP65 (Zschimmer & Schwarz) were filled into in a metal vessel (250 cm^3 volume) and molten in a heating cabinet at 100 °C. Vacuum homogenization of the powder and the binder took place in a heatable dissolver (DISPERMAT©, Getzmann) at a temperature of 100 °C; a stirrer of 40 mm diameter was used. The highest speed of rotation was 1600 rpm.

Rheological behaviour was measured in a plate–plate system (PP25) of a rotary rheometer (MCR 300, Paar-Physica) with an adjusted gap of 0.5 mm. The measurement temperature as given by DoE was adjusted by the peltier element. Before each measurement, a waiting period of 3 min was maintained to achieve temperature equilibrium over the volume of the sample. Feedstocks were measured under shear stress control in a range of stress up to 3000 Pa. Rheological behaviour in DoE was interpreted in terms of the relative viscosity at a shear rate of 100/s. The yield point was ascertained by determining the intersection of two tangents at the point of change from elastic to visco-elastic behaviour [22].

3. Results and discussion

3.1. Determining the centre point

To determine the ranges of this DoE, the following assumption was made: a powder mixture of $ZrSi_2$ and ZrO_2 with a well-established Zr/Si ratio of 0.80 [21] has a rather high theoretical powder density of approx. 5.16 g/cm^3 and a theoretical sintering density of approx. 4.39 g/cm^3. Under these conditions, a powder volume content of approx. 60.8 vol.% is required to achieve net-shape sintering. Considering the use of 7.1 vol.% sintering additives (5.0 wt.% Al_2O_3 and 0.5 wt.% MgO), which are necessary to sinter these materials at adequate

temperatures of about 1575 °C, the theoretical powder density decreases to 5.07 g/cm^3 as well as the theoretical sintering density (4.29 g/cm^3). Thus, a powder volume content of 61.3 vol.% is required. Typically approx. 98% of the theoretical density will be achieved, which means, the required powder content to achieve zero shrinkage is reduced as well.

In the frame of DoE, it has to be ensured that all compositions can be well prepared and the rheological behaviour can be reliably investigated. So in a first definition, the Zr/Si ratio of the center point was fixed at 0.80 in connection with a required powder volume content of 57.5 vol.%. Preliminary experiments showed, that feedstocks with a powder content much higher then 60 vol.% are difficult to prepare and their rheological behaviour is not reliably measurable at all times. So the powder content was chosen to be in range of 55–60 vol.%.

Also the rheological behaviour was not measurable below temperatures of 74 °C for a feedstock composition with a Zr/Si ratio of 0.80 and a powder volume content of 60%. Thus the center temperature was fixed at 100 °C, and the minimum and the maximum for the cube were defined at 85 and 115 °C.

These preliminary tests (see Fig. 1) had also shown that the flowability and the yield point of the feedstock is significantly influenced by using a blend of 50 wt.% Siliplast LP13 and 50 wt.% Siliplast LP65. Mainly Siliplast LP13 is recommended for powders with $d_{50} > 1$ μm. These tests also showed that feedstocks solely based on LP65 have a lower viscosity than feedstocks based on the described binder blend. However, using a binder mixture of the two was found to increase the yield point of the feedstock, which is very important for contour accuracy during debinding. Because of the rather high viscosity of this composition, the amount of Siliplast LP13 in the blends for DoE was significantly reduced to 7.5 wt.% for the center point. The minimum of the Siliplast LP13 content was preset at 5 wt.% and the maximum at 10 wt.%.

Because the increase in volume can be influenced by the Zr/Si ratio, the minimum and the maximum for the cube of this parameter were fixed at 0.70 and 0.90, respectively.

3.2. Computing the design

The experiments were designed with the modde 8.0 (Umetrics Europe, Sweden) software tool. A four-factor design

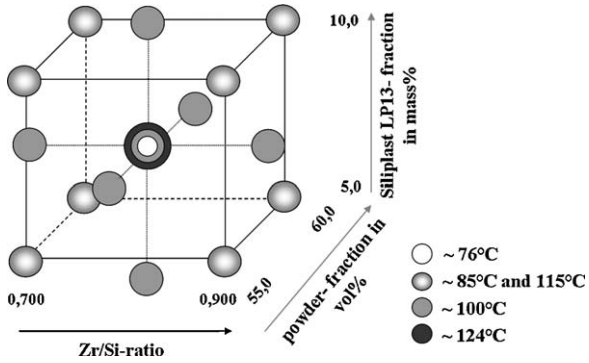

Fig. 2. CCC-model for experimental designs with a star distance of 1.66.

was chosen to study the influence of powder volume content, binder composition, Zr/Si ratio, and temperature on the rheological behaviour of feedstocks for reaction-bonding zircon ceramics. The minimum level of the feedstocks was specified at a powder content of 55 vol.%, a Zr/Si ratio of 0.7, a LP13 fraction of 5.0 wt.%, and a temperature of 85 °C; the maximum level was accordingly: 60 vol.% powder content, 0.9 Zr/Si ratio, 10.0 wt.% LP13 fraction, and 115 °C. The full fractional CCC-model (**C**entral **C**omposite design **C**ircumscribed) studied with a star distance of 1.66 is shown in Fig. 2. Thus, the content of powder ranges from 53.5 to 61.5 vol.%, the Zr/Si ratio from 0.64 to 0.96, the LP13 fraction from 3.5 to 11.5 wt.%, and the temperature from 76 to 124 °C. The volume content of sinter additives due to the purposive variation of the volume ratio of ZrO_2 and $ZrSi_2$ was between 6.3 and 7.4 vol.%.

This CCC-model resulted in a design matrix of 27 experiments. The center point has been replicated three times to detect the deviation of the experimental results. The order of execution of the experiments was completely randomized and given by the software.

3.3. The experimental design

3.3.1. Analysis

Table 2 gives an overview of the experiments studied, their run-order and their measured rheological behaviour. The viscosities observed in the design of the experiments covered a range between 2.7 and 20.6 Pa s. In terms of the lowest viscosity of 2.7 Pa s the temperature is high and the Zr/Si ratio,

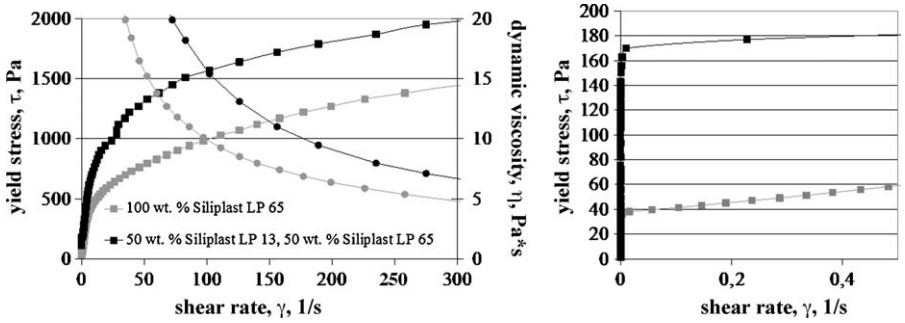

Fig. 1. Comparison of the flowability and viscosity of 55 vol.% powder in Siliplast LP65 and in a 50:50 mixture of LP13 and LP65 (left graph). Detail of the flow curve, highlighting the distinctly increased yield point by using a 50:50 mixture of both binders (right graph).

Table 2
Experimental design matrix.

Influencing variables					Measured character	
Run-order	Zr/Si ratio	Powder content, vol.%	Temperature, °C	Siliplast LP13 fraction, wt.%	Dyn. viscosity, η, Pa s[a]	Yield point, τ_0, Pa
20	0.7	55	85	5	3.9	92
23	0.9	55	85	5	5.7	172
10	0.7	60	85	5	10.7	208
6	0.9	60	85	5	20.6	1050
19	0.7	55	115	5	2.7	82
13	0.9	55	115	5	4.5	160
26	0.7	60	115	5	5.2	78
17	0.9	60	115	5	12.5	582
14	0.7	55	85	10	5.3	135
16	0.9	55	85	10	8.0	320
9	0.7	60	85	10	10.2	305
25	0.9	60	85	10	14.0	540
12	0.7	55	115	10	3.7	110
11	0.9	55	115	10	5.2	216
7	0.7	60	115	10	6.9	225
5	0.9	60	115	10	8.2	150
3	0.64	57.5	100	7.5	3.1	83
21	0.96	57.5	100	7.5	7.0	178
24	0.8	53.5	100	7.5	3.7	135
15	0.8	61.5	100	7.5	18.0	605
4	0.8	57.5	76	7.5	8.2	230
22	0.8	57.5	124	7.5	4.6	177
27	0.8	57.5	100	3.5	5.9	190
8	0.8	57.5	100	11.5	5.2	170
2	0.8	57.5	100	7.5	5.4	192
1	0.8	57.5	100	7.5	6.1	180
18	0.8	57.5	100	7.5	5.8	185

[a] Shear rate 100/s.

the powder content, and the LP13 fraction of the feedstock are low. In contrast to this expected result, a feedstock with a low LP13 fraction surprisingly shows the highest viscosity. Either there has to be an interaction between the LP13 fraction and one or more other factors or the influence of LP13 is insignificant. Similarly this applies to the yield points that range between 78 and 1050 Pa. The following extensive analysis of the DoE gives a more detailed answer.

Table 3 shows the statistic quality factors of the fitted model based on the original data. The coefficient of correlation R^2 and the reproducibility are excellent, whereas the model validity exhibits very low values. The model quality of DoE can be significantly improved by logarithmic transformation of the measured values for both properties. Logarithmic transformation of the results is advisable, if the connections can be seen as multiplicative [23]. An indication for this is a range larger than one order of magnitude, i.e. $y_{max}/y_{min} > 10$, covered by the results. However, there was no legitimate way to change the model validity of the yield point from negative to positive. The model quality factors of the viscosity values after transformation are also mentioned in Table 3.

Table 3
Model quality before and after logarithmic transformation.

	Dyn. viscosity		Yield point	
	Orig.	Transf.	Orig.	Transf.
R^2	0.948387	0.951547	0.840875	0.789495
Q^2	0.714136	0.895624	0.418424	0.532595
Model validity	0.184321	0.527383	−0.2	−0.2
Reproducibility	0.994757	0.98798	0.999204	0.997401

Table 4 lists the main impacts on the rheological parameters, non-significant effects are printed in italics. The first three main effects on both parameters are identical: powder volume content, Zr/Si ratio, and temperature. The powder content and the Zr/Si ratio increase (positive value) viscosity and yield point, while temperature as the third main effect, decreases them (negative value).

Further on, a quadratic term of the powder content effects an increase of the viscosity (fourth rank) as well as of the yield point (fifth rank). Yet, the influence of the quadratic term of powder content on yield point is not significant.

DoE analysis, however, revealed an unexpected viscosity-decreasing interaction of the powder volume content and the Siliplast LP13 fraction as well as an interaction of Siliplast LP13 with the Zr/Si ratio. LP13 was used in this DoE in the range between 3.5 and 11.5 wt.% in order to increase the yield point of compositions with rather low dynamic viscosities. A pronounced yield point is of high importance for thermal debinding of samples to avoid shape distortion.

Table 4
Main impacts on the rheological behaviour of the studied feedstocks.

Dyn. viscosity	Effect	Conf. int (±)	*Yield point*	Effect	Conf. int (±)
Powder content (PC)	0.3603	0.0517	Powder content (PC)	0.3272	0.1349
Zr/Si ratio	0.2043	0.0517	Zr/Si ratio	0.3227	0.1349
Temperature	−0.1823	0.0517	Temperature	−0.1793	0.1349
PC^2	0.1510	0.0670	PC^2	*0.1728*	*0.1748*
PC × Siliplast LP13	−0.0917	0.0594	PC × Siliplast LP13	*−0.1182*	*0.1550*
Zr/Si ratio × Siliplast LP13	−0.0638	0.0594	Zr/Si ratio × Siliplast LP13	−0.1748	0.1550
Siliplast LP13	*0.0137*	*0.0517*	Siliplast LP13	*0.0346*	*0.1349*

However, this could not be realized as expected. The interaction of the Zr/Si ratio and Siliplast LP13 content acts more significantly on the yield point than the interacting effect of the contents of powder and Siliplast LP13 does. But both interacting terms were found to be marginal and, moreover, to lower the yield point.

LP13 regarded on its own has no significant effect on the viscosity or the yield point. The effect of decreasing viscosity can hardly be explained by means of the particle size distribution (psd). Increasing the Zr/Si ratio should shift the psd to smaller diameters as a consequence of the higher ZrO_2 content in the powder mixture and accordingly the specific surface of the powders to higher values. With the increase of the Zr/Si ratio the tap density and hence their compactibility should change as well. Table 5 shows the measured values for the powders, but there is no significant tendency noticeable, neither for the specific surface nor for the tap density in dependence of the Zr/Si ratio. This might be caused by the influence of the suspension viscosity during the wet-milling of the powder mixtures for 24 h. Preparing the powder mixtures at the same solvent to powder ratio, thus discounting the suspension viscosity, may have an impact on the efficiency of the wet-milling process in a way, that the resulting powder characteristics are not comparable.

At least increasing the powder volume content should also increase the surface area to be wetted by the binder. Yet, the viscosity-decreasing interaction of the powder volume content and the Siliplast LP13 fraction is not evident. An explanation for these effects may be the different specific density of the used binders as well as their different thermal expansion behaviour, which was observed by measuring the density as a function of temperature (Table 6). The thermal expansion behaviour of the binders influences the amount of the powder volume content of the feedstock dependant on the temperature. Calculating the powder volume content at 120 °C for a feedstock with Siliplast LP65 showed that it would have a 0.3% higher powder content than a feedstock based on Siliplast LP13. Especially at high powder contents little variations in powder content have a high impact on the rheological behaviour of feedstocks. While calculating the compositions for DoE, the binder's densities have been assumed as equal, as a density of 0.9 g/cm^3 was specified by the manufacturer for both binders.

Table 5
Specific surface (BET) and tap density of powder mixtures.

Zr/Si ratio	Specific surface (BET), m^2/g	Tap density, % T.D.
0.64	13.3	27.0 ± 0.39
0.70	14.0	26.5 ± 0.17
0.80	14.5	27.0 ± 0.08
0.90	14.3	25.7 ± 0.10
0.96	14.3	26.5 ± 0.63

3.3.2. Optimization

Optimization was performed on the basis of this analysis. The software calculates the process and composition data to achieve the required quality. Optimization served to minimize viscosity and maximize the yield point. As both conflicting properties cannot be optimized in a single composition, two compositions were calculated by the software. The recommended parameters and the predicted rheological values of these feedstocks are shown in Table 7. Both feedstocks were prepared and characterized in line with specifications of the optimization tool.

The predicted rheological behaviour was achieved for a feedstock with a Zr/Si ratio of 0.744. The measured viscosity was 3.9 Pa s at a shear rate of 100/s, and a yield point of approximately 230 Pa was found (Fig. 3). Compared to the predicted values of 4.3 Pa s for the viscosity and 183 Pa for the yield point, the difference to the experimental values is acutely low. This is also true for the composition with a Zr/Si ratio of 0.925, where a viscosity of 24.3 Pa s and a yield point of approximately 460 Pa were determined.

The measured data prove the high validity of the DoE investigated and evaluated. The differences to the predictions may be due to specific surface effects. A different batch of the intermetallic compound, $ZrSi_2$, was used for the optimization of these two compositions. A specific surface of 5.7 m^2/g was

Table 6
Density of the binders as a function of temperature.

Temperature, °C	$\rho_{\text{Siliplast LP13}}$, g/cm^3	$\rho_{\text{Siliplast LP13/LP65 (7.5 wt.%/92.5 wt.%)}}$, g/cm^3	$\rho_{\text{Siliplast LP65}}$, g/cm^3
20	0.906	0.921	0.921
60	0.788	0.799	0.802
70	0.782	0.792	0.795
80	0.777	0.785	0.790
90	0.768	0.779	0.781
100	0.762	0.771	0.775
110	0.755	0.769	0.766
120	0.749	0.752	0.760

Table 7
Composition parameters and predicted properties of optimized feedstock.

Zr/Si ratio	Powder content, vol.%	Temperature, °C	Siliplast LP13 content, wt.%	Dyn. viscosity, Pa s	Yield point, Pa
0.744	53.5	121	11.5	4.3	183
0.925	61.2	124	5.2	17.0	673

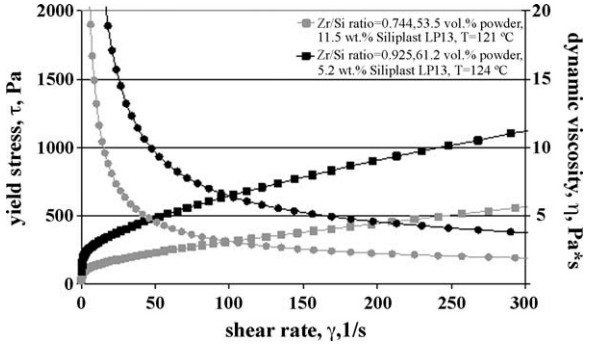

Fig. 3. Rheological behaviour of feedstocks produced in line with specifications of the DoE optimization tool.

measured for this batch instead of 6.7 m^2/g for the former batch. Nevertheless, prediction and optimization operations on the basis of this DoE are valid. This paper shows that design of experiments is very well adapted for materials development. The main advantage of this operation method, beside the economy of time and materials, is the high benefit in information content at comparable low effort as shown in Fig. 4. The figure gives an overview of the rheological behaviour in dependence of the varied factors of DoE as well as marking the range of compositions with zero shrinkage. The black area identifies the typical working range for hot-moulding processing up to 4.0 Pa s and the grey area shows the viscosity range up to 20.0 Pa s, which is well suited for low-pressure injection moulding. With increasing temperature and increasing Siliplast LP13 content the compositions become more suitable for shaping via hot-moulding technology. Increasing the powder content and the Zr/Si ratio renders them more adequate for low-pressure injection moulding. But the whole investigated area is useful for thermoplastic shaping with paraffin based feedstock compositions. Compositions between the two inserted lines for densities of 95 and 100% T.D. enable sintering with complete compensation of shrinkage.

As mentioned before, zero shrinkage compositions can be well calculated but because of variances of the starting materials, especially in parameters like their specific mass change and/or their specific density, shrinkage behaviour has to be finally proved by experiment.

The results of this investigation give a good overview on rheological properties of feedstocks for the reaction-bonded net-shape ceramics and offer the possibility to tailor the compositions in a wide range depending on the desired characteristics of the feedstock and eventually also enables an adjustment of the sintered microstructure as will be shown in a subsequent paper.

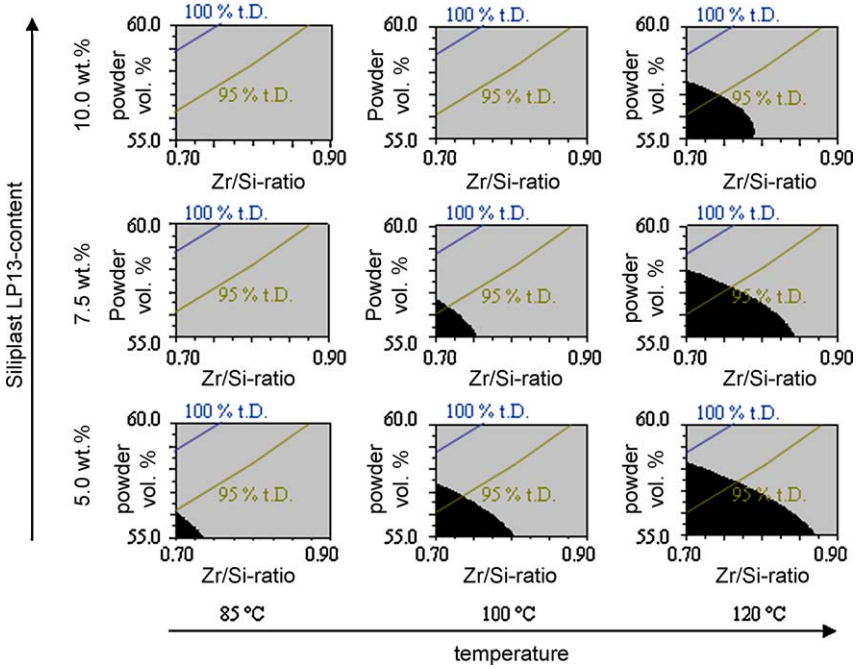

Fig. 4. Overview on feedstock viscosities depending on the varied factors of DoE. The black areas indicate the typical working range for hot-moulding processing up to 4.0 Pa s, and the grey areas show the viscosity range up to 20.0 Pa s, well suited for low-pressure injection moulding. (The lines that indicate a sinter density of 95 and 100% T.D., respectively, were only added for clarification.)

4. Conclusions

Within the framework of a DoE, a range of compositions were studied, which can be processed by low-pressure injection moulding with dynamic viscosities between 2.7 and 20.6 Pa s. The main influencing factors in this study are the powder volume content, the Zr/Si ratio, the binder composition, and the temperature, as well as the interactions of the binder LP13 and the powder content or the Zr/Si ratio, respectively. Evaluation of the experimental data showed the need for logarithmic transformation of the responses in order to achieve reasonable model quality factors. Analysis of this transformed model revealed unexpected effects of the Siliplast LP65/Siliplast LP13 ratio due to the not considered difference in the density of the two binders. In the end, the validity of this DoE was confirmed by two feedstocks optimized towards low viscosity and a high yield point, respectively, as specified by the software.

The study and calculations of the required powder content demonstrated that low-pressure injection moulding of reaction-bonded net-shape zircon ceramics is feasible with various compositions, depending on specific requirements of rheology and/or sintering shrinkage. In addition to the possibility of net-shape sintering, negative or positive shrinkage of a ceramic body is adjustable as well. Finally, besides the shrinkage behaviour, the microstructure of the ceramic can also be modified, which will be reported in a subsequent paper. In summary, the combination of a flexible and economical processing technology, such as low-pressure injection moulding, with a reaction-bonding net-shape material, such as the zircon ceramic described, is technically feasible and highly attractive to produce ceramic devices in microtechnology.

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