JOURNAL OF MATERIALS SCIENCE 24 (1989) 840–848

The control of sprue solidification time in ceramic injection moulding

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The ability to control the sprue solidification time by using modulated pressure was investigated over a wide range of mould temperatures using a silicon nitride suspension. The mould temperature needed for infinite sprue solidification time was reduced from 158 to 75° C by the application of a pressure of 132 MPa modulated at 0.5 Hz. Under similar moulding conditions, macroscopic moulding defects in the hub of a solid rotor were avoided. Such defects were present in all static pressure mouldings. The thermal power input to the sprue resulting from oscillatory flow was estimated.

1. Introduction

The production of ceramic injection mouldings containing sections thicker than 10 to 15 mm on a reproducible scale is at an early stage of development [1]. The major reason is the tendency for these moulded bodies to contain internal voids or cracks arising from the shrinkage of the polymer–ceramic suspension in the mould [2].

The effect of these defects on the mechanical properties of the final component is catastrophic. During cooling in the mould, the sprue solidifies and prevents the static hold pressure from filling the cavity and compensating for shrinkage. Thus, a sealed pocket of fluid at the centre of sections thicker than the sprue solidifies and shrinks in isolation.

Recent literature has described a solution to this problem which has been applied successfully to unfilled polymers in the first instance [3–5] and subsequently to short fibre reinforced thermoplastics [6]. Recently, this technique has been successfully extended to ceramics [7, 8]. It involves the application of oscillating rather than static pressure to the fluid during the solidification stage. This is achieved by incorporating an oscillating pressure device mounted between the machine nozzle and the mould (Fig. 1). Such a device suitable for abrasive ceramic blends has been designed [9] and its performance demonstrated [10]. The device is able to modulate the pressure between wide limits for a selected duration with a preset frequency and background pressure.

The resulting oscillatory flow in the sprue dissipates heat which gives rise to a prolonged solidification time for the material in this section of the mould. The additional input of material, which compensates for the shrinkage of the moulding, could continue for as long as required, provided the central region of the sprue remains molten.

Recent research [8] has indicated that, under modulated pressure moulding conditions, sprue solidification time increased monotonically with oscillating pressure amplitude, but the correlation was dissimilar for different compositions and mould temperatures.

TABLE I Composition of the sinterable silicon nitride powder

Constituent	Density ($kg\,m^3$)	wt %
Silicon nitride	3190	84.97
Y_2O_3	5010	5.94
Al_2O_3	3987	4.83
SiO_2*	2200	4.26

*Includes SiO_2 present on Si_3N_4.

The aim of the present work was to assess the influence of mould temperature on sprue solidification time for mouldings produced both under static hold pressure and oscillating pressure and to determine how this affected the final product. Silicon nitride powder was used in conjunction with a polypropylene–microcrystalline wax organic vehicle to produce a straight bladed rotor (Fig.2). A rotor with a solid hub was used, rather than the component prepared in previous work [8] because it corresponds more closely to projected designs. This shape was selected as a low-cost substitute to simulate the problems of injection moulding a turbo-charger rotor. The reasons for selecting the polypropylene-based organic vehicle have been described elsewhere [11–13].

2. Experimental details

2.1. Ceramic powder

Silicon nitride powder (from Anzon, UK) was used after milling with sintering aids (Table I). The particle size distribution of the milled powder is shown in Fig. 3.

2.2. Formulation

The silicon nitride powder was compounded with the organic components using a twin screw extruder that gave good dispersion [14]. The method has been

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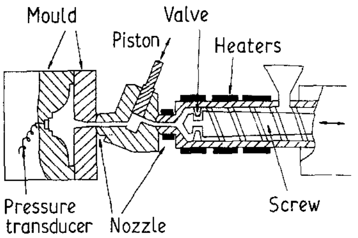

Figure 1 Schematic representation of the oscillating pressure valve and other moulding equipment.

described elsewhere [11–13]. Details of the machine conditions are given in Table II. The composition is given in Table III. The ceramic volume loading was 50% being the maximum that would give suitable viscosity for this particular powder blend and milling condition.

2.3. Injection moulding

A Bone Cravens Daniels 350-120 injection moulding machine was employed and the operating conditions are given in Table IV. For the oscillating pressure valve, a frequency of 0.5 Hz and a compression pressure of 132 MPa were selected in preliminary experiments with this cavity and material and were used throughout this work. The relaxation and background pressure were kept constant at 52 and 30 MPa, respectively. The former is the pressure needed to retract the piston while the latter is the pressure applied by the screw to feed the piston chamber.

The mould temperature was varied between 20 and 70° C and mouldings were made both under static hold pressure and oscillating pressure. Mouldings were also made under static hold pressure at a mould temperature of 80° C. The pressure on material during the injection stroke was set at 152 MPa in each case and this pressure was also used as the static hold pressure.

All mouldings were visually examined and those without external flaws were contact radiographed using an HP Faxitron X-ray system operating at 50 kV and 3 mA in order to detect internal flaws. Some rotors were then sectioned along the axis for further visual examination.

3. Results and discussion

3.1. Static pressure moulding

Figs 4a–e show examples of cavity pressure–time traces of mouldings made using static hold pressure. The curve in Figs 4c and e shows that the cavity transducer registered a constant pressure approximately up to the sprue solidification time. Thereafter, the full pressure could not be transferred to the moulding. The cavity pressure traces (Figs 4a, b and d) did not all show this feature fully. The initial hold and therefore the sprue freeze off time were sometimes masked by fluctuations soon after injection. This was caused by audible slip-stick motion of the screw in the barrel as pressure was transferred to the cavity.

TABLE II Compounding machine conditions

Screw diameter (mm)	40
Screw L/D ratio	17
Screw speed (r.p.m.)	60
Barrel temperatures (° C)	220(feed)-225-235-225(nozzle)

TABLE III Composition of injection moulding blend

	wt %
Silicon nitride	78.01
Polypropylene	14.65
Microcrystalline wax	4.89
Stearic acid	2.45
Vol % silicon nitride	50

A computer simulation model [15] was used to determine the sprue solidification time at different mould temperatures. The model [15] considers one-dimensional heat transfer analysis with finite difference approximation using the Crank–Nicolson algorithm. The finite difference approximation results in a system of Equations [15] represented by:

$$A_i T^*_{i-1} + B_i T^*_i + C_i T^*_{i+1} = D_i \qquad (1)$$

$$i = 0, 1 \ldots n - 1, n \qquad (2)$$

$$A_0 = 0 \quad \text{and} \quad C_n = 0 \qquad (3)$$

where n refers to the number of layers into which the sprue section is divided, i is the layer number and T^* is the temperature after a selected time interval dt. The values of A, B and C are dependent on the thermal diffusivity of the formulations, dt and the distance between adjacent layers. The value of D depends on these as well as the mould temperature. At the mould wall, the values of A and D depend on the heat transfer coefficient which was taken as $1000\,\mathrm{W\,m^{-2}\,K^{-1}}$ [15]. The system was solved on computer and the temperature at the centre of the sprue was calculated after each time interval dt. This temperature was compared with the softening point of the formulation which was 158° C [2]. When the centre temperature of the sprue was equal to the softening point $\Sigma\, dt$ was reported as the sprue solidification time. Clearly, the effect of undercooling and also the influence of pressure on melting point modify the temperature at which solidification occurs. For these compositions, it has been shown that these affects approximately cancel for this organic blend [2]. The material properties used in the computer simulation are given in Table V. For the calculation of composite properties, only silicon nitride at 50 vol % and polypropylene were considered. The rule of mixtures was used to calculate the density and the specific heat. The average value of the Hashin and Shtrikman [19] upper and lower bound was used as the thermal conductivity. The justification for use of these rules to calculate the composite properties has been discussed elsewhere [8].

TABLE IV Injection moulding machine conditions

Screw diameter (mm)	45
Screw L/D ratio	18
Screw speed (r.p.m.)	45
Barrel temperature profile (° C)	160(feed)-225-230-230-225(nozzle)
Oscillating pressure valve temperature (° C)	225

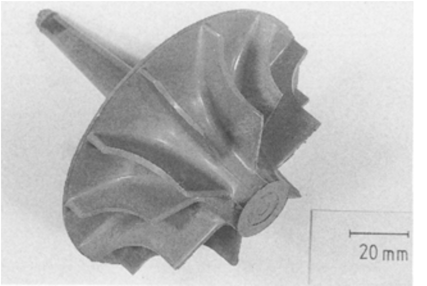

Figure 2 The straight-bladed rotor.

3.2. Modulated pressure moulding

For mouldings made with pressure modulation, examples of the cavity pressure–time traces obtained are shown in Fig. 4f–i. Oscillating pressure was introduced immediately after injection. The shape of these traces can be explained as follows. The lower bound of the latter part of the trace (shown as CX in Fig. 4i) can be extrapolated to the point of injection I (Fig. 4i). The trace IX thus constructed is similar in form to the pressure–time curve for mouldings made under static hold pressure. However, only section CX of this notional curve is real. Point C represents the solidification of the sprue for reasons described below. In section CX, the lower bound represents the fraction of residual pressure resulting from the moulding operation which was registered on the cavity pressure transducer at the mould wall after sprue solidification. This decayed with time, as expected. Superimposed on this curve in the region CX are pressure fluctuations arising from the fact that the oscillator was still active after the sprue solidified. Clearly, the moulding was able to transmit some of the pressure and the oscillation could continue indefinitely if required, but with little advantage for the quality of moulding [8].

In the region IC where the sprue was molten, the cavity pressure transducer initially registered the full pressure fluctuations arising from the preset maximum and minimum settings on the oscillating valve. As the suspension cooled in the cavity, the full pressure from the valve was not transmitted to the transducer and, therefore, the upper envelope decayed with time. As point C was approached sprue solidification was almost complete and pressure transmission on the compression stroke fell steeply. Similarly, the release of pressure from the cavity on the decompression stroke was attenuated and the two envelopes converged. Thus, point C represents the final solidification of the central channel of the sprue and enables the sprue solidification time to be read directly from the pressure–time trace.

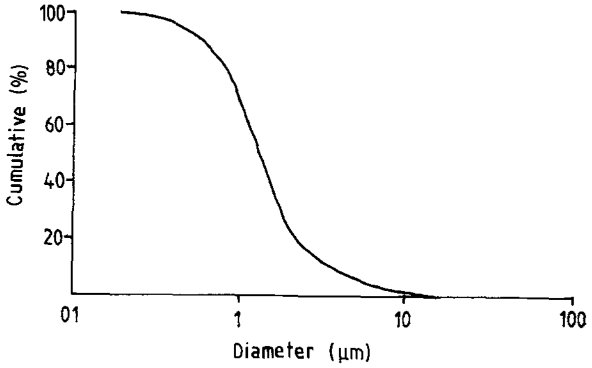

Figure 3 Particle size distribution for the milled silicon nitride powder.

3.3. Sprue solidification time

Fig. 5 presents graphs of sprue solidification time against mould temperature for mouldings made with static hold pressure and oscillating pressure. For mouldings made with static hold pressure, sprue solidification times shown were calculated using the model [15]. The sprue consisted of a cone 60 mm in length with a semi-angle of 3.7°. The nozzle end had a radius of 4.29 mm. This end cannot be taken as the location of initial sprue solidification because of axial heat flow from the nozzle of the moulding machine. Using a graphical method of multiple dimension unsteady state heat flow [20], the point at which axial heat flow was insignificant was found to be a distance of two diameters (17.16 mm) along the sprue and the radius at this point was 5.4 mm. This radius was taken for calculations of sprue solidification time using the model with $n = 10$ and $dt = 0.1$. In fact, the effect on calculated solidification time of using this slightly larger radius was very small.

Solidification times at 90, 100, 120, 140, 150 and 155° C were also calculated in order to construct the full curve although mouldings were not made at these temperatures. As expected, the model predicts that the sprue solidification time is infinite when the mould temperature is equal to the softening point of the formulation. Also, at 60 and 80° C under static hold pressure the sprue solidification times which could be determined from the cavity pressure–time trace as discussed before, were 10 and 12 sec, respectively. These times compare well with 7.9 and 8.9 sec predicted by the model considering that the model predicts a lower bound for the sprue solidification time and that assumptions were made in determining the material properties used. The graph also shows that under static hold pressure a 60° C increase in mould temperature (from 20 to 80° C) could only effect an

TABLE V Properties of component materials and the formulation used for the computer simulation

Material	Density ($kg\,m^{-3}$)	Specific heat ($J kg^{-1} K^{-1}$)	Thermal conductivity ($W m^{-1} K^{-1}$)	Thermal diffusivity ($m^2 sec^{-1} \times 10^{-6}$)
Silicon nitride	3229*	710 [16]	17.2 [18]	-
Polypropylene	905†	1923 [17]	0.12 [17]	-
Formulation	2067‡	1317‡	3.72§	1.37

*Calculated from Table I.
†Grade GY545M, e.g. ICI Ltd, Welwyn Garden City, UK.
‡Rule of mixtures.
§Average of Hashin and Shtrikman [19] bounds.

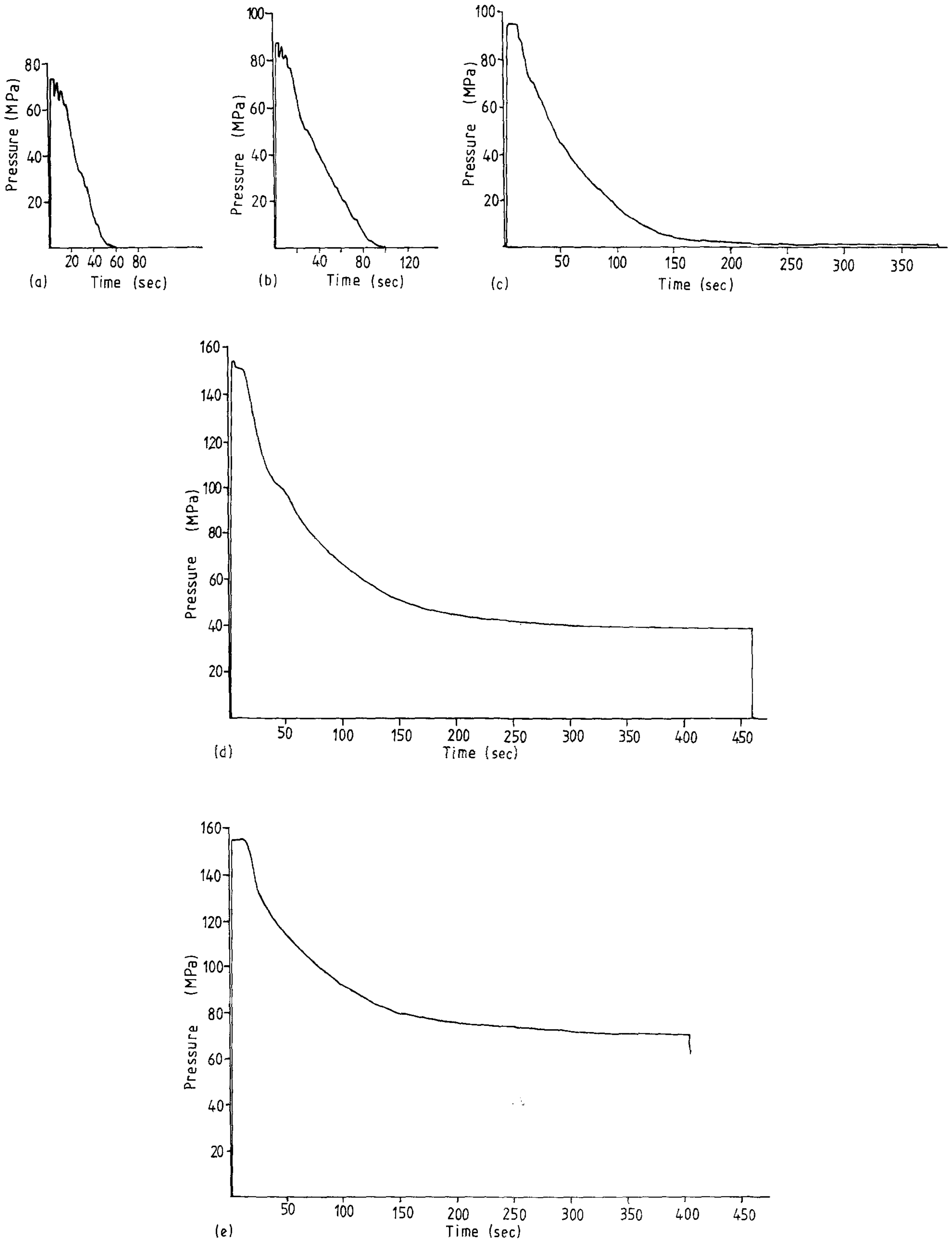

Figure 4 Cavity pressure–time traces for mouldings made: (a) static hold pressure and 20° C mould temperature; (b) static hold pressure and 50° C mould temperature; (c) static hold pressure and 60° C mould temperature; (d) static hold pressure and 70° C mould temperature; (e) static hold pressure and 80° C mould temperature; (f) oscillating pressure and 20° C mould temperature; (g) oscillating pressure and 50° C mould temperature; (h) oscillating pressure and 60° C mould temperature; (i) oscillating pressure and 70° C mould temperature.

increase of 2.3 sec in sprue solidification time and that a mould temperature close to the softening point of the formulation would be necessary to increase the sprue solidification time significantly. Nevertheless, it is not practical to increase the mould temperature to such values because of binder degradation which causes blistering in the mouldings and also mould ejection problems associated with deformation of the moulding [21].

For the mouldings made using oscillating pressure, the sprue solidification time was measured from cavity pressure–time traces as discussed in section 3.2. The graph of sprue solidification time against mould temperature (Fig. 5) is similar in shape to the curve for

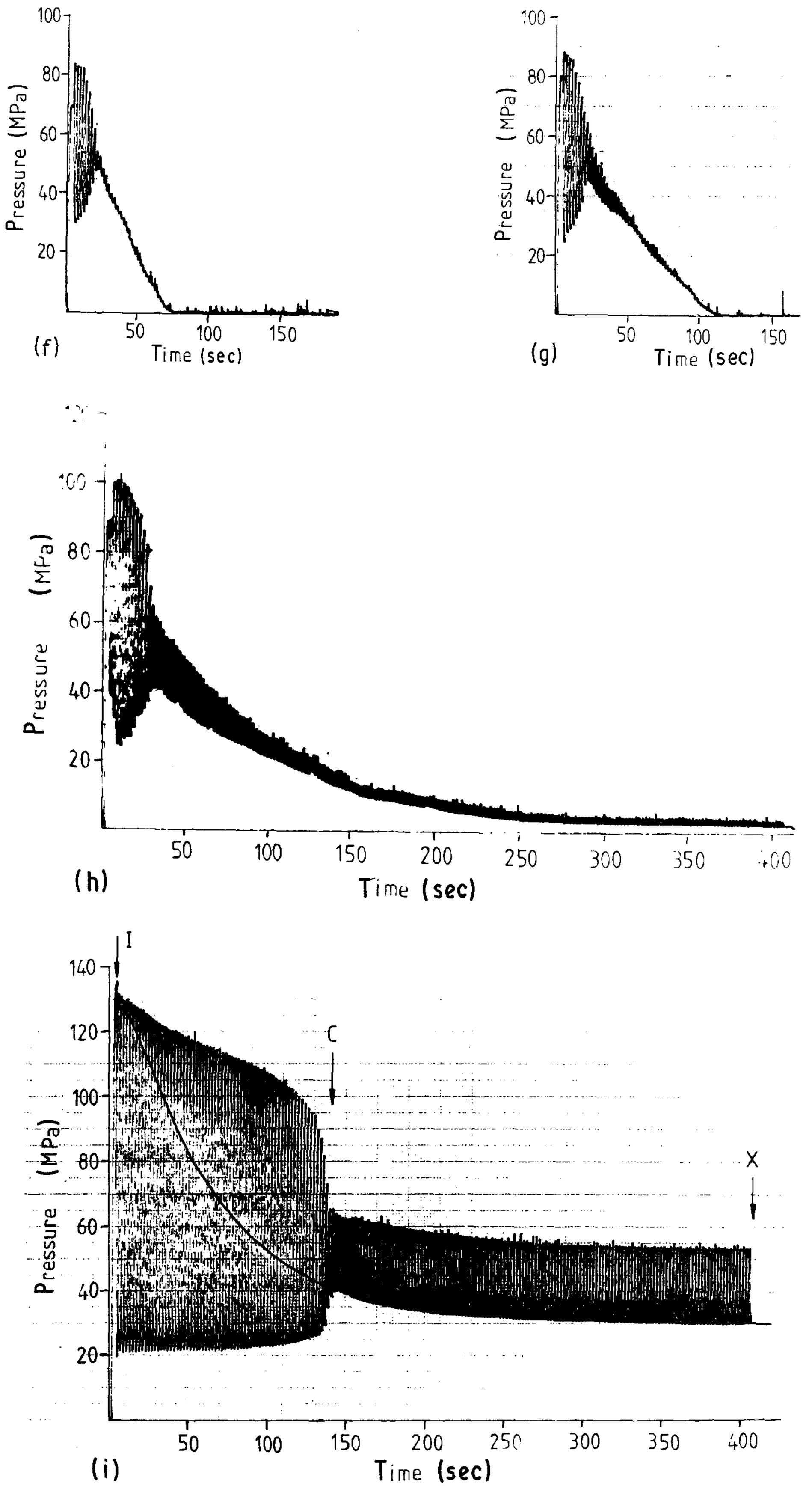

Figure 4 Continued

the static hold pressure condition but is displaced to longer times because heat dissipated in oscillating flow increased the sprue solidification time. The graph when extrapolated shows that at ~75° C, the sprue solidification time becomes infinite. This is because at ~75° C mould temperature, the heat input in the sprue due to a combination of oscillatory flow and adiabatic compression is equal to the heat flow across the mould walls surrounding the sprue.

3.4. Calculation of sprue heat input

It is possible to estimate the heat input to the sprue for

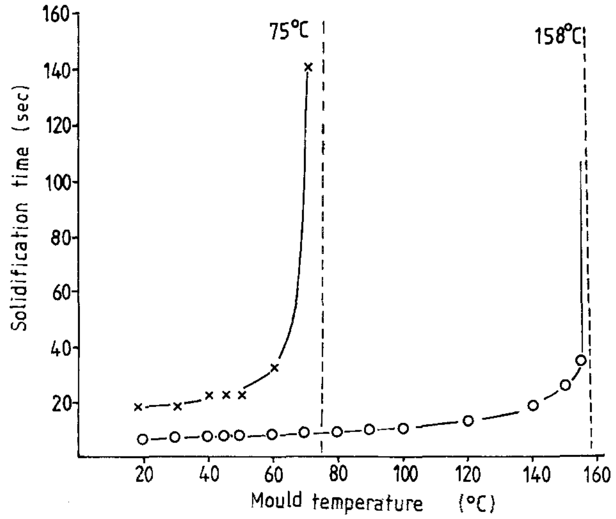

Figure 5 Graph of sprue solidification time against mould temperature for mouldings made using static hold pressure and oscillating pressure.

TABLE VI Sprue power inputs for different molten zone sizes

r_i (mm)	Sprue power input (W)
0.1	30
0.5	50
1.0	70

the steady state condition where thermal input near the centre equals heat flow rate across the solidified cylindrical layer. This value for an infinite cylinder, q ($\mathrm{W\,m^{-1}}$) can be found from:

$$q = \frac{(T_s - T_m)}{\dfrac{l_n r_o/r_i}{2\pi k} + \dfrac{1}{2\pi r_o h}} \qquad (4)$$

where k is the thermal conductivity of the suspension, taken as $3.72\,\mathrm{W\,m^{-1}\,K^{-1}}$, h is the heat transfer coefficient taken as $1000\,\mathrm{W\,m^{-1}\,K^{-1}}$, T_s is the solidification temperature taken as 158° C and the mould temperature T_m is 75° C by construction of the asymptote in Fig. 5. Also, r_o is the sprue radius which was 5.4 mm and r_i is the radius of the molten channel. Since the sprue was 60 mm in length, the power input for this particular mould and suspension can be found for different values of r_i and these values are tabulated in Table VI. Although r_i cannot be found directly from the present experiments, the values given cover a range of one order of magnitude and give rise to a sprue input power of 30 to 70 W. In fact, the thermal resistance offered by surface heat transfer to the mould wall was negligible for $h = 1000\,\mathrm{W\,m^{-1}\,K^{-1}}$. The range of r_i chosen is considered adequate for transmission of pressure to the moulding.

It is not claimed that the benefit in terms of moulding quality described in secion 3.6. is attributable only to the additional thermal energy input. The effect of oscillatory flow may also be beneficial in, for example, destroying weld lines or modifying internal stresses. The above discussion reveals that there is a mould temperature which allows the sprue to be kept molten indefinitely and if static hold pressure were used this temperature would be the softening point of the formulation which was 158° C in this instance. On using oscillating pressure, the mould temperature required to keep the sprue molten indefinitely is ~75° C, almost 80° C less than the corresponding value for the static pressure situation.

3.5. Cavity pressure

Fig. 6a shows graphs of maximum cavity pressure, expressed as a percentage of the pressure on material applied by the screw which was 152 MPa, as a function of mould temperature for mouldings made using static hold pressure. Fig. 6b shows the maximum cavity pressure during the oscillating pressure region expressed as a percentage of the applied amplitude which was 132 MPa, again as a function of mould temperature. The maximum cavity pressure recorded during injection moulding was in the region of 150 MPa (100%) at 80 and 70° C mould temperature when using static hold pressure, indicating that pressure loss in the material in the nozzle, sprue and mould cavity was very low at these mould temperatures. Similarly, in the oscillating pressure case, the cavity pressure registered the full applied pressure amplitude of 132 MPa at a mould temperature of 70° C.

The cavity pressure recorded depends on the pressure transferred through the material in the sprue and moulding and is related to its viscosity at low shear

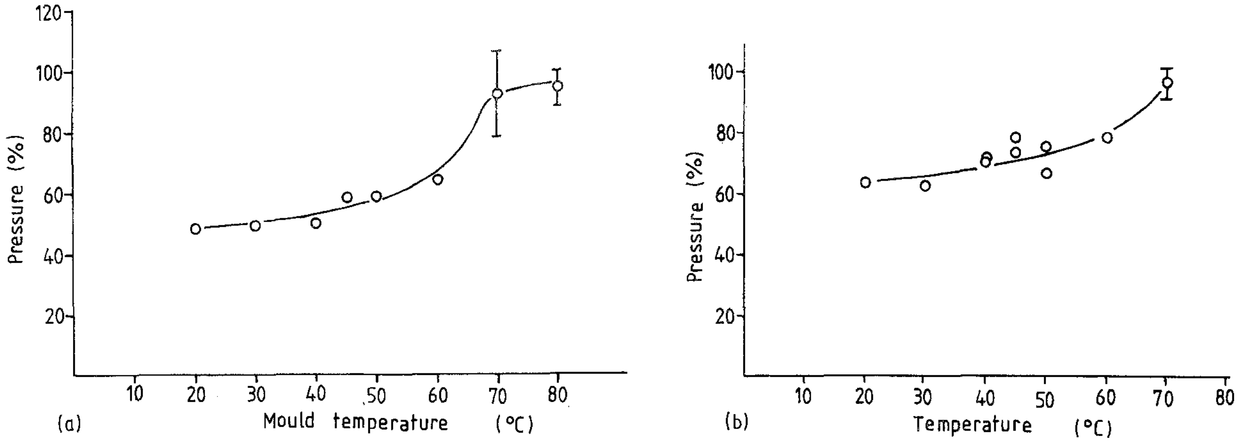

Figure 6 (a) Measured maximum cavity pressure as a percentage of applied pressure on material at injection as a function of mould temperature. (Applied pressure on material = 152 MPa.) (b) Measured maximum cavity pressure as a percentage of applied pressure amplitude for the pressure modulated region of solidification. (Applied pressure amplitude = 132 MPa.)

rates. The nominal injection speed was 61 $cm^3 sec^{-1}$ based on an empty shot. In practice, the presence of viscous material can extend the mould filling time considerably. Thus, although, the volume of each rotor was 70.3 cm^3, the mould filling time may be > 1 sec and this can be seen from the pressure trace although the scale precludes precise measurement. Thus, chilling of the material in the sprue and at the mould wall adjacent to the pressure transducer reduced the efficiency of pressure transmission and this effect can be related to mould temperature as shown in Fig. 6.

A related effect can be seen at the end of each moulding cycle. In Figs 4d and e, corresponding to static pressure moulding conditions with 70 and 80° C mould temperatures, respectively, the cavity pressure did not return to zero until the hold pressure applied by the screw was released. This residue is not a measure of pressure in the moulded body, but is rather a fraction of the applied hold pressure transmitted through the solid moulding to the cavity pressure transducer situated directly opposite the sprue. A similar behaviour is seen in Fig. 4i for modulated pressure moulding at a mould temperature of 70° C. In this case, the pressure decayed to the background pressure of 30 MPa. The background pressure is the hold pressure applied by the screw to keep the oscillating valve chamber fully charged. At temperatures < 60° C, pressure transmission through the solid moulding was inefficient and the residual pressure effect was not observed.

Although the softening point of the formulation was 158° C, it is interesting to note that the organic fraction of the blend contains 22 wt % wax with a melting point of 60° C. Differential scanning calorimetry (DSC) showed that the organic components crystallise as separate phases [2] and therefore, the efficiency with which the solid suspension transmits pressure may be related to incipient melting of the wax component.

3.6. Integrity of mouldings

Table VII describes the macro defects observed in mouldings made at different mould temperatures with and without the use of oscillating pressure. For mould temperatures from 20 to 40° C, all mouldings contained cracks that were detected by visual examination. The intensity of cracking for mouldings made with static hold pressure and oscillating pressure gradually decreased as higher mould temperatures were reached, and at 40° C, with oscillating pressure, only fine cracks were visible at the web–sprue junction as shown in Fig. 7.

Between mould temperatures of 45 to 60° C, radiography revealed that mouldings made with both static and oscillating pressure contained cracks (Table VII). At a mould temperature of 70° C, radiographs showed that the mouldings made using static hold pressure contained cracks at the centre and at the web–sprue junction of the rotor. These observations were confirmed on sectioning a rotor made using these conditions (Fig. 8a).

The use of oscillating pressure at a mould temperature of 70° C made it possible to produce rotors without macro defects. Radiography revealed that macro cracks or voids were absent and this was confirmed by visually examining a section (Fig. 8b).

An attempt was made to produce mouldings at a mould temperature of 80° C using static hold pressure with the aim of obtaining macro defect-free components without the use of oscillating pressure. Radiography revealed that cracks were present at the centre and the web–sprue junction of the moulding and this was confirmed on examining a section (Fig. 8c).

These observations on the mouldings can be explained by referring to the deductions made on mould temperature, sprue solidification time and cavity pressure. Under static hold pressure, up to 80° C mould temperature, the mouldings suffered from macro defects because sprue freeze-off was too rapid. Therefore, the static pressure was unable to continue to fill the mould cavity and compensate for shrinkage. The use of oscillating pressure did not enhance the sprue solidification time sufficiently until the suitable mould temperature (70° C) was reached. At this mould temperature the sprue freeze-off time was 140 sec, ~4.5 times the corresponding value at 60° C. In addition, the graph of sprue solidification time against mould temperature (Fig. 5) suggests that at about 75° C mould temperature the sprue solidification time can be increased indefinitely. Therefore, the conditions at 70° C mould temperature provide effective packing of the moulding with the ceramic suspension over a long period and the ability to compensate

TABLE VII Defects observed in rotor mouldings

Mould temperature (° C)	Quality of mouldings	
	Static hold prsesure	Oscillating pressure
20	macro cracks on web	macro cracks on web
30	macro cracks on web	macro cracks on web
40	macro cracks on web	cracks at web–sprue junction
45	cracks at the centre alongside blades	cracks at the centre alongside blades
50	cracks perpendicular to axis at the web–sprue junction	cracks perpendicular to axis at the web–sprue junction
60	fine crack at the centre alongside blades	cracks perpendicular to axis at the web–sprue junction
70	cracks at the centre and web–sprue junction	macro defect-free
80	cracks perpendicular to axis at the centre and web–sprue junction	not tested

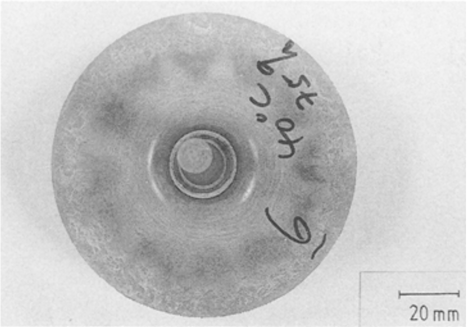

Figure 7 Moulding made at 40° C mould temperature using oscillating pressure.

for shrinkage is enhanced. This is reflected in the macro defect-free mouldings obtained.

4. Conclusions

A ceramic blend with 50 vol % silicon nitride powder and with polypropylene as the binder was injection moulded into a rotor-shaped mould cavity. Moulding was performed under static hold pressure, and with the use of oscillating pressure at a frequency of 0.5 Hz and a pressure amplitude of 132 MPa applied immediately after injection, at different mould temperatures.

A simulation model, previously verified, was used to calculate the sprue solidification times for mouldings made using static hold pressure, as it was not always possible to estimate this from cavity pressure–time traces. Where this was possible, there was close agreement between measured and calculated values. Correct interpretation of the oscillating pressure–time traces allowed the sprue solidification time to be read directly from the curves.

For the static pressure moulding situation, a mould temperature equal to the softening point of the suspension was needed to achieve infinite sprue solidification time. By using modulated pressure, the corresponding mould temperature was reduced from 158 to 75° C. Under these conditions, the heat input caused by oscillatory flow in the sprue could be estimated at 30 to 70 W for this mould cavity and suspension. Mould temperature also had a pronounced effect on peak injection cavity pressure; the full value being obtained only for mould temperatures of 70 and 80° C.

The correct choice of oscillating pressure conditions and mould temperature allowed the solid hub of the rotor to be fabricated without macroscopic defects. All mouldings made with static pressure showed cracking apparently resulting from shrinkage-related stresses. The absence of such cracks is related to the extension of sprue solidification time allowing efficient packing at the centre of the hub as a result of oscillatory flow.

Acknowledgements

The authors are grateful for SERC support for this ceramic injection moulding programme. BCRL supplied the sinterable silicon nitride powder. Mr G. Ragbir kindly performed the radiography.

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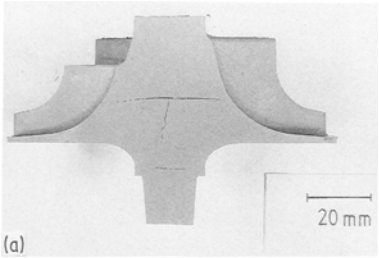

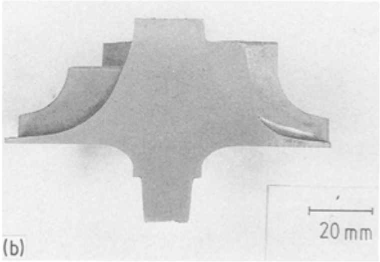

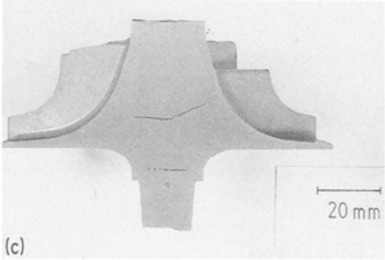

Figure 8 Sections of mouldings made with: (a) Static hold pressure and 70° C mould temperature; (b) Oscillating pressure and 70° C mould temperature; (c) Static hold pressure and 80° C mould temperature.

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Received 18 February
and accepted 25 May 1988