



# Ceramic core–shell composites with modified mechanical properties prepared by thermoplastic co-extrusion

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## Abstract

Bi-layer rods of core–shell geometry were prepared by thermoplastic co-extrusion of assembled feedrods. The zirconia-toughened alumina was used for the tough core and alumina for the hard shell surrounding the core. Core–shell rods with three different shell thicknesses were successfully co-extruded and regular interface between the core and the shell was achieved. The benefits of using the shell feedstock with a lower viscosity compared to the viscosity of the core feedstock were described and discussed in detail. The perfect joining between the core and the shell components achieved during co-extrusion was retained also after binder removal and sintering. The component interface was sharp and relatively smooth. The core–shell structure resulted in a modification of the mechanical properties of bi-layer rods in comparison with monolithic rods. Flexural strength tests of monolithic and bi-layer sintered rods were performed and the advantage of the core–shell concept was presented and explained.

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## 1. Introduction

Monolithic (single-component) ceramics do not always meet all mechanical and/or other functional properties required for a final product. Composite ceramic materials are therefore being developed and increasingly utilized. The bi-layer structure with core–shell geometry is a simple composite system that can provide parts with modified mechanical as well as functional properties. Core–shell composites can be prepared in the shape of axially symmetric structures as rods or tubes, where a shell (outer layer) surrounds concentrically a core (inner layer). The shell and the core are produced either of different ceramic materials or of the same materials but with a different porosity [1]. Such

structures can be used as oxygen separation membranes [2,3], solid oxide fuel cells [4,5] or bioceramic implants with a bone structure [6–8]. The mechanical properties of the core–shell composite can be advantageously modified in order to utilize the composite in applications where monolithic ceramics fail, e.g. for a part where hard and wear resistant surface is required while retaining the tough and high-strength behaviour of the core [9].

There are many methods for ceramic core–shell part production, e.g. electrophoretic deposition [10], extrusion or slip casting followed by dip-coating [1,11], freeze casting [7], and co-extrusion [12]. Each method has its own advantages and limits in the production of core–shell structures. The advantages of co-extrusion include the principle simplicity, perfect joining of the core and shell materials and the low-cost production. There are two approaches to the production of core–shell structures by the co-extrusion method; multi-billet (multichannel) extrusion [13] and piston extrusion of an assembled thermoplastic preform called feedrod [12,14]. In both cases, the rheological

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behaviour of the ceramic feedstocks plays a key role in obtaining the required geometry of the co-extruded product without any irregularities and defects. Ceramic thermoplastic feedstocks usually have non-Newtonian flow behaviour that can be satisfactorily described (in the shear rate range of interest) by the two-parameter power-law model (the Ostwald–de Waele relationship) [15,16]:

$$\tau = K \times \dot{\gamma}^n, \quad (1)$$

where  $\tau$  is the shear stress,  $K$  reflects the consistency index of the feedstock (the higher values represent more viscous materials),  $\dot{\gamma}$  is the shear rate, and  $n$  is the power-law index giving a measure of the pseudoplasticity (for  $n < 1$  showing shear-thinning behaviour). The shear stress ( $\tau_w$ ) and the apparent shear rate ( $\dot{\gamma}_{app}$ ) at the wall in a capillary nozzle can be calculated as [17]

$$\tau_w = \frac{R \Delta p}{2 L} \quad (2)$$

and

$$\dot{\gamma}_{app} = \frac{4Q}{\pi R^3}, \quad (3)$$

where  $\Delta p$  is the pressure drop between capillary ends,  $Q$  is the volumetric flow rate, and  $L$  and  $R$  are the length and radius of the capillary, respectively. Eq. (3) is only valid for Newtonian liquids. For non-Newtonian liquids, the Weissenberg–Rabinowitsch correction must be applied to the apparent shear rate. For a power-law liquid, the non-Newtonian shear rate is given by [15,17]:

$$\dot{\gamma}_w = \frac{4Q}{\pi R^3} \frac{3n+1}{4n}. \quad (4)$$

The velocity profile of flow in a capillary is parabolic for Newtonian liquids. For liquids that fulfil the power-law, the velocity profile can be expressed as [18]

$$v(r) = \left( \frac{\tau_w}{KR} \right)^{1/n} \frac{n}{n+1} \left[ R^{n+1/n} - r^{n+1/n} \right], \quad (5)$$

where  $r$  is the radial position. After combining Eq. (5) with (1) and (4) and rearranging we obtain:

$$v(r) = \frac{Q(3n+1)}{\pi R^2(n+1)} \left[ 1 - \left( \frac{r}{R} \right)^{n+1/n} \right]. \quad (6)$$

The velocity profiles in a capillary nozzle calculated at the same volumetric flow rate for various power-law indexes,  $n \leq 1$ , are shown in Fig. 1. We can see the increasing plug-like nature of the flow with the decreasing power-law index, which means that just a thin layer close to the capillary wall is effectively sheared. With the co-extrusion of two or more components, the flow becomes more complex. Fortunately, ceramic co-extrusion can advantageously utilize extensive experiments and analyses performed on polymers. Several authors have described and analysed flow instabilities in multilayer co-extrusion that can result in an irregular interface and intermixing of layers [18–20] or even in the encapsulation phenomena in which the polymer with a lower viscosity is wrapped around the polymer with a

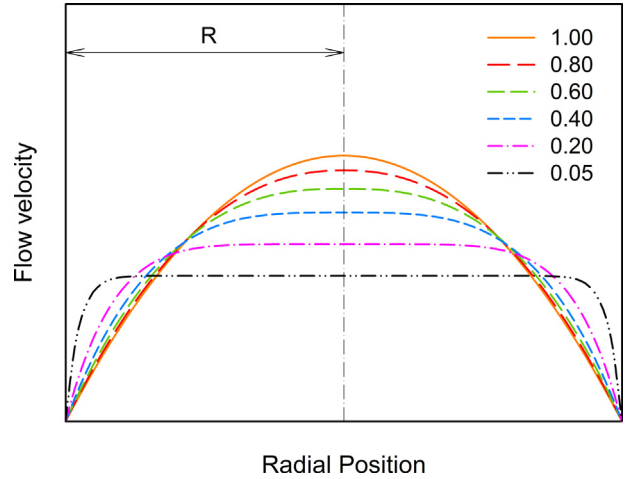


Fig. 1. Velocity profiles of laminar flow in a capillary nozzle for various exponents of power-law liquids calculated for the same volumetric flow rate.

higher viscosity [18,21–23]. For the case of core–shell extrusion through a capillary nozzle the velocity profiles in the shell and the core have to be calculated separately for each component as [18]

$$v_s(r) = \left( \frac{\tau_w}{K_s R} \right)^{1/n_s} \frac{n_s}{n_s+1} \left[ R_i^{n_s+1/n_s} - r^{n_s+1/n_s} \right] \quad (7)$$

and

$$v_c(r) = \left( \frac{\tau_w}{K_c R} \right)^{1/n_c} \frac{n_c}{n_c+1} \left[ R_i^{n_c+1/n_c} - r^{n_c+1/n_c} \right] + \left( \frac{\tau_w}{K_s R} \right)^{1/n_s} \frac{n_s}{n_s+1} \left[ R_i^{n_s+1/n_s} - R_i^{n_s+1/n_s} \right], \quad (8)$$

where the subscripts  $s$  and  $c$  stand for the shell and core, respectively, and  $R_i$  is the position of the interface between the shell and the core. Because the position of the core–shell interface is not known a priori, it must be found experimentally or predicted from the total volumetric flow rate. The total volumetric flow,  $Q$ , may be obtained using Eqs. (7) and (8) as

$$Q = Q_s + Q_c = 2\pi \int_{R_i}^R v_s r dr + 2\pi \int_0^{R_i} v_c r dr, \quad (9)$$

where  $Q_s$  and  $Q_c$  are volumetric flow rates of the shell and the core component, respectively. After integrating we obtain:

$$Q = \left( \frac{\tau_w}{K_s} \right)^{1/n_s} \left( \frac{\pi R^3 n_s}{3n_s+1} \right) \left[ 1 - \left( \frac{R_i}{R} \right)^{3n_s+1/n_s} \right] + \left( \frac{\tau_w}{K_c} \right)^{1/n_c} \left( \frac{\pi R^3 n_c}{3n_c+1} \right) \left( \frac{R_i}{R} \right)^{3n_c+1/n_c}. \quad (10)$$

The position of the core–shell interface,  $R_i$ , can be calculated numerically, when the total volumetric flow rate, wall shear stress and power-law parameters of the core and shell components are specified.

The characterization of the rheological behaviour of the extruded materials was determined as one of the most

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