Preparation and Rheological Investigations on Thermoplastic ZrO_2 Pastes

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Introduction

Thin ceramic tubes with a wall thickness less than 300 µm are interesting for various applications, for example as protective layer for a plastic shaft, filter structures, thermowells, and heat exchangers. Advantages are higher wear and lower filtration resistance, or faster heat transfer.

For producing such tubes, water based duroplastic or thermoplastic binder systems can be used. The main problem with water-based binders is the collapsing of the tubes after extrusion.

Extruded duroplastic compounds cannot be recycled whereas thermoplastic binders can be recycled and do not collapse after extrusion.

To optimize the ceramic thermoplastic compound for the tube production, ZrO_2 powder, Hostamont and polyethylene were mixed in different percentage by volume.

Experimental Procedure

First, fine zirconia powder (TZ-3YE, Tosoh Europe B.V.) was kneaded in a torque rheometer mixer (Rheomix 600, Gebr. Haake GmbH) with Hostamont (EK 583, Clariant GmbH) and LDPE (Elf-Atochem AG). After kneading, the torque was measured three times with 5 rpm, at 160°C. The material was fed in a screw extruder (Rheomex 202, Gebr. Haake) and characterized by capillary rheometry at 160°C. Tubes were produced with the screw extruder and a die (d_a = 6.7 mm, d_i = 6.2 mm).

Figure 1 shows zirconia powder, plastic compound and a sintered ceramic tube.

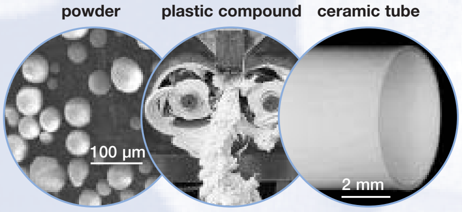

Fig. 1: *Material in different process stages*

Compound Preparation

Figure 2 shows the results of the kneading process for different compounds.

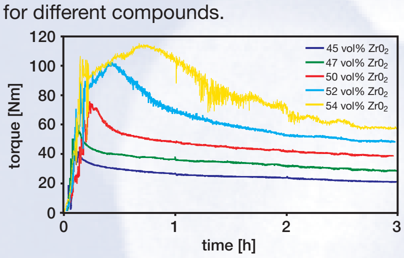

Fig. 2: *Kneading results of different compounds*

Rising the amount of ZrO_2 powder, the torque increases, and getting a steady state takes a longer time.

Table 1 presents the color and the consistency after kneading. In the compounds with more than 50 vol.-% ZrO_2 powder the color changed after mixing because of abrasion.

vol.-% ZrO2	color	cold consistency
45	beige	plastic paste
47	beige	plastic paste
50	beige	plastic granulate
52	grey	plastic granulate
54	grey	plastic granulate

Tab. 1: *Compound properties after kneading*

Torque Rheometer Test

Internal mixers used by industry as well as torque rheometers are designed to create as much turbulent flow as possible to provide optimum mixing for heterogeneous compounds. Torque rheometers are not designed to define the absolute viscosity of the compounds, but it makes a good sense to apply a certain rotor speed and to measure the reactionary torque on the rotors. Two batches for all different compounds were kneaded. After kneading, the torque was measured three times with 5 rpm, at 160 °C, for all compounds. For a stable process the torque should always be the same. Figure 3 shows the result of the test.

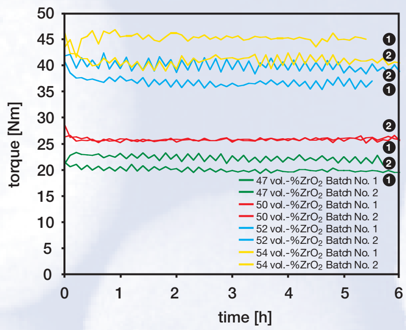

Fig. 3: *Torque for different compounds (5 rpm, 160°C)*

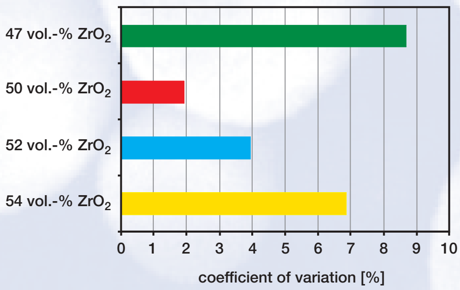

Fig. 4: *Comparison of the coefficient of variation*

The lowest coefficient of variation between two charges was reached with a compound of 50 vol.-% ZrO_2 powder.

Capillary Rheometry Test

For heterogeneous material it is very difficult to define the absolute viscosity, because the homogeneity can change during the test. Therefore, only the pressure between the different compounds is compared. All compounds, except 54 vol.-% ZrO_2, could be extruded. Figure 5 shows the pressure during the tests with the different compounds.

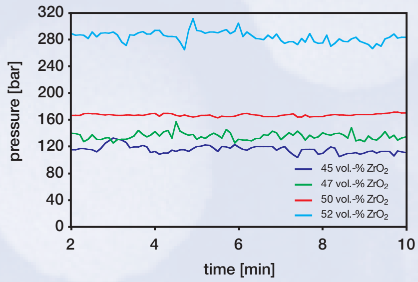

Fig. 5: *Pressure for different compounds during the test (20 rpm, at 160 °C)*

The compound with 50 vol.-% ZrO_2 is the most homogenous material.

Tube Production

Tubes were produced with all compounds. Table 2 shows some visual properties seen after extrusion.

vol.-% ZrO_2	color	roundness	pressure
45	beige	-	not stable
47	beige	-	not stable
50	beige	+	stable
52	grey	+	not stable

Tab. 2: *Visual properties after extrusion*

In Figure 6 sintered tubes with a wall thickness of 200 µm are depict.

Fig. 6: *Tubes after sintering*

Conclusions

With the compound having 50 vol.-% ZrO_2 the best results concerning homogeneity of pastes and extrudability were reached. Table 3 shows the results.

	pressure
torque rheometer test	reproducible batches
capillary rheometry test	stable pressure
tube production	beige color stable process

Tab. 3: *Results of compound 50 vol.-% ZrO_2*

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