

Flowability and Moldability of Zirconia Micro-Part Via Micro-Powder Injection Molding

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ABSTRACT

The micro-powder injection moulding process is a favourable process to produce micro-sized components of zirconia. The aim of this study is to study the flowability of zirconia feedstock and to ensure the moldability of micro-sized zirconia parts without any defects. To obtain the critical powder volume percentage (CPVP), zirconia was deposited inside a Brabender mixer while oil was added gradually with an interval of three minutes. The value of 2,3 and 4 vol.% less than CPVP was mixed with palm stearin and low-density polyethylene (LDPE) binders to produce three feedstocks and run through a rheometer to test the flowability of each feedstock. The feedstock with the best flowability characteristic was then subjected to micro-powder injection moulding process through trial and error to achieve the optimum parameters for micro-sized zirconia part without any defects. The CPVP of zirconia achieved for this study was 49.92 vol.% in which the optimal loadings of 46 vol.%, 47 vol.% and 48 vol.% feedstocks were analysed using the rheometer. The results show that 46 vol.% feedstock was most suitable for producing zirconia micro-part without any defects compared to other powder loadings due to its low viscosity, high shear thinning, flow behaviour index less than 1 and low activation energy. Injection moulded zirconia micro-sized green parts without any defects were produced using the following parameters: 10 bar of injection pressure, 70 °C of mould temperature, 190 °C of melt temperature and 7 seconds of injection time, respectively. Zirconia micro-sized green parts regularly exhibited short-shot defects when mould temperatures less than 70 °C were applied. The results showed that by employing feedstocks containing 46 vol.% of zirconia, micro-sized zirconia parts without any defects can be produced successfully through the micro-powder injection moulding process.

Keywords: Zirconia, Binder System, Optimal Loading, Rheology, Micro-Powder Injection Moulding

INTRODUCTION

Zirconia is a suitable material for dental applications due to its biocompatibility, structural integration, mechanical properties and low solubility (Daou 2014). Zirconia oxide (ZrO_2) is the only metal oxide that has acid, alkali and also the ability to reduce and oxidize (Sesli et al. 2018). Zirconium oxide (ZrO_2) shows three polymorphs; monoclinic, tetragon and symmetrical cube. The monoclinic phase is stable at room temperature and changes to the tetragonal phase at 1170°C during heating (Nikiforov et al. 2015). Monoclinic zirconia exists below a temperature of 1240°C and is stable in the room temperature phase of

pure zirconia. While tetragonal zirconia is an intermediate phase, which exists between temperatures of 1240°C and 2370°C. Retention of the tetragonal phase can be done if cubic zirconia is added with a dopant (Miller et al. 2013). In this study, Zirconia will be subjected to powder metallurgy process that is powder injection moulding (PIM).

The powder injection moulding process, (PIM) is able to produce products that are small in size and have a complex and complicated surface structure (Arifin et al. 2014). This process uses powder metallurgy technology where it compresses fine-sized powder into the desired shape after the sintering process (Mohamad et al. 2020).

This high-tech process is widely applied in various industries such as automotive, medical, electrical components that require product shapes with complex geometry (Quinard et al. 2009). This process also does not require secondary processes such as casting, grinding and machining to obtain the final product (Ismail et al. 2012). Therefore, this PIM process saves costs in addition to being practical through mass production (Gonzalez-Gutierrez et al. 2012). This technology has been used since the late 70's when the demand for products with micro surface characterization was increasing year after year (German & Bose 1997). This is due to its ability to produce products that approach the final form as desired.

The micro powder injection moulding process, μ PIM has similarities with PIM where the stages and types of processing are the same, only the μ PIM process produces products with a micro-sized design (Fayyaz et al. 2015). There are four important processes that must be carried out before obtaining the final body with the best mechanical properties.

The entire PIM process starts from mixing, injection moulding, solution or thermal debinding and finally the sintering process to obtain the final body with the best mechanical properties (Ibrahim et al. 2010). The powder applied in this PIM technology is made of ceramics depending on the characteristics and properties of the desired final part (Salleh et al. 2017). Feed material in the form of granules is prepared when the powder is mixed with the binder system during the first processing, which is mixing (Gülsoy et al. 2016). Various mixing methods can be used with appropriate velocities (rounds per minute). The rheological property test is performed on the feed material to study the flow properties and obtain the best flow from the rheological aspect (Agote et al. 2001). Phenomena such as the separation of powder-binding materials capable of producing defects can be known (Basir et al. 2021).

The second processing stage is injection moulding where the feed material is melted and pushed into the mould cavity to produce the desired product (Ahn et al. 2009). The successfully injected product is known as the green part. There are various parameters involved in the injection moulding process that can affect the green part such as physical defects. Parameters such as temperature, pressure and time are closely related in producing a green part with zero defects (Basir et al. 2020). Short shots, sparks, weld lines, wrinkles and cracks are common types of physical defects (Basir et al., 2023). Successfully injected green parts go through a debinding process to remove the binder system before the part is sintered (Thian et al. 2001). It can be done in stages; solution or thermal. The temperature used during the debinding process is above the melting temperature of the binder (Torralba et al., 2013). Products

with binding material that has been successfully removed are known as brown parts. It is very fragile and breaks easily so careful handling should be practiced (Ukwueze et al. 2018).

The final part that has gone through the sintering process will experience shrinkage due to recrystallization and compression when it is heated close to the melting temperature of the powder and held for several hours (Nor et al. 2018). The shrinkage of the final part depends on the degree of porosity and porosity of the final body when the binder system is removed (Zakaria et al. 2021). The binding material that used to fill the pores in the interstices and between the powders creates pores and pores as they are removed during the debinding process (Lin & Hwang 2010). The powder will go through the process of grain growth in filling the existing pores and pores, then the grain will touch each other on the grain border with each other (Mohd et al. 2013). The sintering process is very important where the mechanical properties can be improved by the sintering cycle being worked on and changed (Aggarwal et al. 2007). A good sintering cycle can produce a fine grain that provides high strength and hardness during mechanical testing (Basir et al. 2021).

Mixing, injection moulding, debinding and sintering are the four important processes that must be carried out before obtaining the final part with the best mechanical properties. In this study, mixing and injection moulding processes were the primary focus, whereby the flowability and moldability of Zirconia feedstock were obtained through three important stages. The first stage is to obtain the critical powder volume percentage (CPVP) (Reddy et al. 1996). Zirconia was deposited inside Brabender mixer while oil was added gradually with an interval of three minutes. For the second stage, The value of 2,3 and 4% less than CPVP was mixed with palm stearin and low-density polyethylene (LDPE) binders to produce three feedstocks and run through a flow tester to test the flowability of each feedstock (Amin et al. 2023). For the last stage, the feedstock with the best flowability characteristic was then subjected to micro-powder injection moulding process through trial and error to achieve the optimum parameters for micro-sized zirconia part. The aim of this study is to study the flowability of zirconia feedstock and to ensure the moldability of micro-sized zirconia parts without any defects.

METHODOLOGY

The material used in this research was 3 mol% yttria-stabilized zirconia (3YSZ) which was obtained from Inframat Advanced Materials LLC in Manchester,

Connecticut, USA. The Horiba Laser Scattering Particle Size Distribution Analyzer LA-960 shows that it has a mean particle size of 1.0410 μm as can be seen in Figure 1(a). The measured pycnometer density of Zirconia was 5.6387 g/cm^3 , obtained from the Quantachrome Ultracycrometer 1000. Table 1 below shows the characteristics of Zirconia material. The field emission scanning electron microscope (FESEM, Zeiss Merlin Compact) was used to inspect the morphology of Zirconia powder and is displayed in Figure 1(b). The elemental composition of Zirconia was investigated with the utilization of the EDX point analysis and EDX layered spectrum analysis as shown in Figure 1 (c). The contents of Zirconia exhibit 67.5 Wt% of Zirconium, 20.8 Wt% of Oxygen and 11.7 Wt% of Carbon.

TABLE 1. The measured characteristics of Zirconia.

Material	Zirconia
Powder median size (μm)	1.0410
Density (g/cm^3)	5.6387
Composition (%)	83

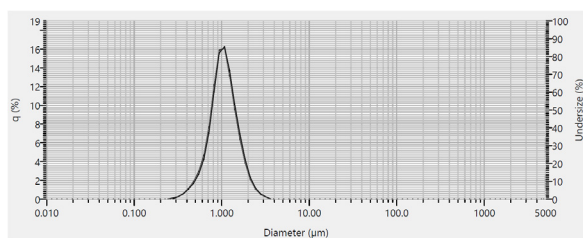


FIGURE 1. (a) Powder median size of Zirconia powder.

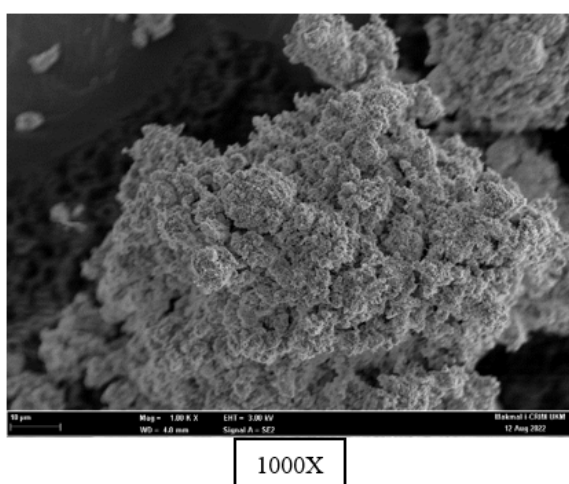


FIGURE 1. (b) Morphology of Zirconia powder.

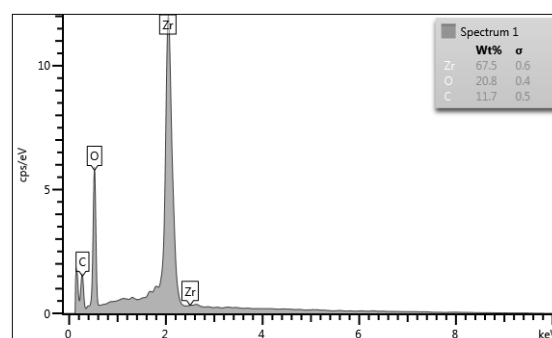


FIGURE 1. (c) EDX graph of Zirconia powder materials.

Using the appropriate binder material, a desired feedstock which has a pseudoplastic flow behaviour can be achieved. The binder system designed for this experiment consisted of low-density polyethylene, LDPE (40 wt%) and palm stearin (60 wt.%). All these binders were supplied by Sime Darby Kempas Sdn. Bhd. and Polyolefin Company (Singapore) Pte Ltd., respectively, had densities of 0.891 and 0.91 g/cm^3 , respectively. Choosing a binder system is important in controlling feedstock homogeneity and injection moulding output quality (Cava et al. 2015; Nor Amalina et al. 2014; Rezvani & Shokuhfar 2012). The mixture with a smaller powder loading has a large surface area, which can increase its viscosity (Kadiman et al. 2018). Table 2 shows the binder properties for LDPE and palm stearin. A critical powder volume percentage (CPVP) analysis shows that the critical powder loading is 49.92 vol.% which follows based on ASTM D281-31 standards. To ensure process flexibility, it is best to choose powder loading values that are 2–5% below the CPVP value.

TABLE 2. Properties of binders

Binder	LDPE	Palm Stearin
Density (g/cm^3)	0.891	0.91
Melting temperature ($^{\circ}\text{C}$)	128.4	45.3
Composition (wt%)	40	60

Therefore, values of 2, 3 and 4 vol.% less than the CPVP values which were 46 vol.%, 47 vol.% and 48 vol.% were chosen as powder loading of the feedstock. The Brabender model W50EHT mixer is used to perform the mixing. During the mixing phase, the binder must be fully melted. Differential scanning calorimetry (DSC) analysis was used to determine the melting point of the binder. To determine the flow of the feedstock produced using a capillary rheometer (Shimadzu CFT-500D), rheological testing based on ASTM D1238 standard was carried out for these three powder loadings. The green part was created using an Xplore IM12 table-top injection moulding

machine by injecting the feedstock into the micro-sized mould on the grounds of the previously studied parameters (Basir et al. 2020) and is shown in Table 3. The dimensions of the micro-sized components of Zirconia are schematically depicted in Figure 2 (a).

TABLE 3. Injection parameters for Zirconia micro-parts.

Injection parameters	Operational process
Injection pressure	10 bars
Mold temperature	70 °C
Melt temperature	190 °C
Injection time	7 s

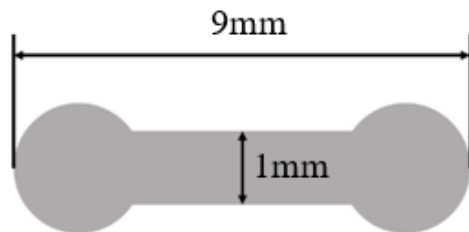


FIGURE 2. (a) Schematic of Zirconia micro-part

According to Newton’s law of viscosity, three types of slopes can be produced from the viscosity against the shear rate graph. The first is a Newtonian slope, in which the line is straight, meaning it is a constant slope which equals to constant viscosity with the increase of shear rate. The other two slopes are non-Newtonian and can be categorized as dilatant (shear-thickening) and pseudoplastic (shear-thinning), in which the line is curvy, as can be seen in figure 3. The desired slope in relation to Viscosity against shear rate in terms of injection moulding are of the pseudoplastic or shear thinning type as it shows good flow behaviour during injection moulding (Thavanayagam et al. 2015). All the graphs of optimal loadings 46, 47 and 48 vol.% of zirconia exhibit pseudoplastic behaviour. according to the same study, Shear viscosity over 1000Pa.s is not commendable as high viscosity and shear rate can cause the separation of powders and binders during the injection process. According to the graphs obtained, the only optimal loading that has a value of viscosity below 1000Pa.s was 46 vol.% of Zirconia. Therefore, it is best suited to be used for the injection moulding process.

RESULTS AND DISCUSSION

The range of optimal powder loadings were determined by the critical powder volume percentage (CPVP). CPVP

is obtained by dripping 1ml of oil every three minutes in brabender with zirconia powder. Figure 2 (b) illustrates the CPVP obtained after an addition of 16 ml of oleic acid. The calculation was made and CPVP obtained was 49.92 vol.%.

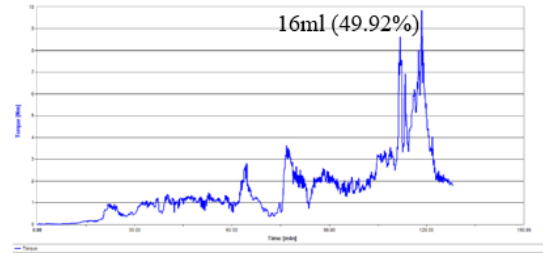
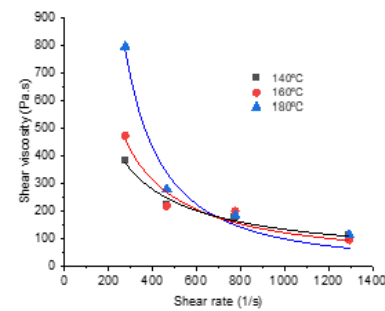
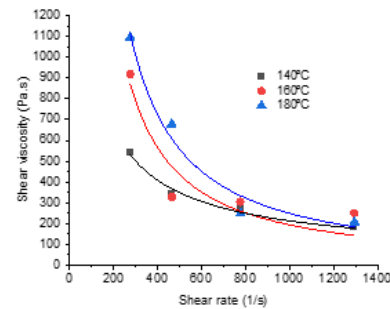


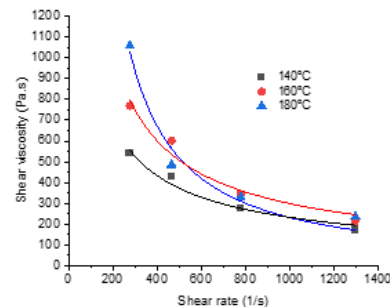
FIGURE 2. (b) Critical powder loading (CPVP) of Zirconia.



(a)



(b)



(c)

FIGURE 3. Graph of viscosity in relation to shear rate of Zirconia, (a) 46 vol.%, (b) 47 vol.% and (c) 48 vol.%

From the CPVP obtained, optimal loadings of 46, 47 and 48 vol.% were chosen to be rheologically analysed with varying temperatures of 140 °C, 160 °C and 180 °C. Viscosity against shear rate values is evaluated through the rheological properties of the Zirconia feedstock, as shown in Figure 3 (a), (b) and (c).

The optimum powder loadings were also analysed from the rheological test and the flow behaviour index and activation energy result of the feedstock at different temperatures of 140°C, 160°C and 180°C were shown in Table 4 and Figure 4 below. The flow behaviour of the feedstock is very important to determine the injectability of the mixture. According to Omar et al. (2020), low viscosity, high strength and good stability are desirable qualities in feedstock. To facilitate the injection moulding process, the PIM feedstock should behave in a pseudoplastic manner where the flow behaviour index, n , is less than 1. The lowest n values calculated from Table 4 were 0.446, 0.434, and 0.442, for temperatures of 140 °C, 160 °C, and 180 °C respectively. The feedstock flows considerably more fluidly when viscosity decreases. Additionally, as the shear rate rises, the feedstock's viscosity likewise reduces. The shear thinning behaviour of the zirconia feedstock is an advantage in the injection moulding process. According to the study by Gülsoy et al. (2016), the PIM feedstock is processed under temperature and pressure so that, as the shear rate increases, the viscosity of the feedstock rapidly decreases. Based on the same study, it is mentioned that the optimal loading with the lowest activation energy is most preferred, as the lower the activation energy, the less power is needed to run the process thereby gaining an advantage over other optimal loadings. As can be seen in Figure 4, the activation energy of powder loading 46 vol.% is the lowest (6.823 kJ/mol) followed by 47 vol.% (47.596 kJ/mol) and 48 vol.% (48.596 kJ/mol). Therefore, the lowest activation energy powder loading of 46 vol.% will be used to proceed powder injection moulding process.

TABLE 4. Values for flow behaviour index and activation energy of Zirconia.

Powder loading (Vol.%)	Temperature (°C)	Flow Behaviour Index (n)	Activation Energy, E (kJ/mol)
46	140	0.446	6.823
	160	0.434	
	180	0.442	
47	140	0.446	47.596
	160	0.469	
	180	0.462	
48	140	0.447	48.596
	160	0.456	
	180	0.460	

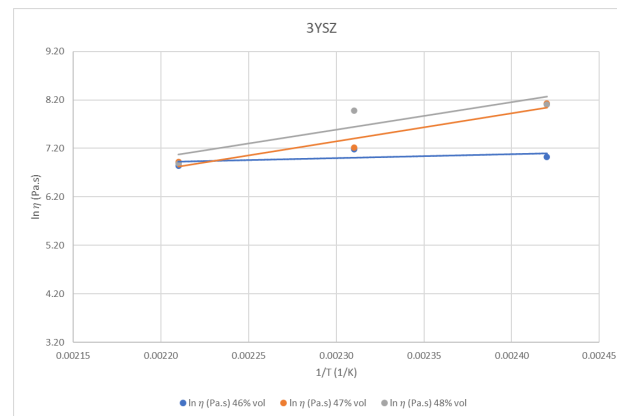


FIGURE 4. Gradient of $\ln \eta$ vs $1/T$ of Zirconia.

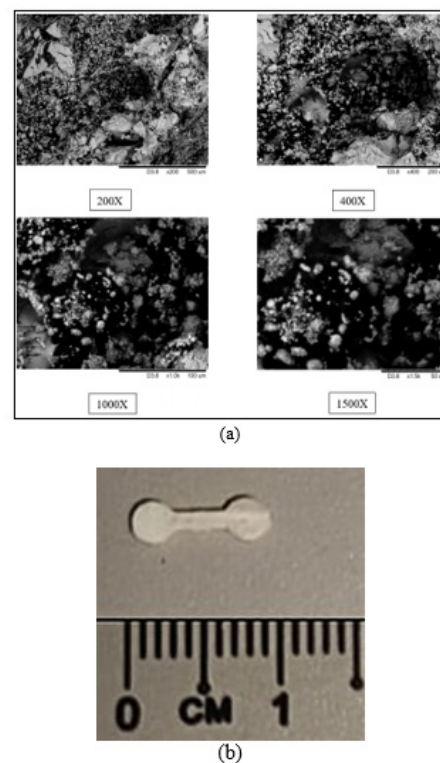


FIGURE 5. (a) SEM images of Zirconia (b) Green micro-part of Zirconia

The scanning electron microscope (SEM) image of the Zirconia feedstock is demonstrated in Figure 5 (a). Based on Figure 5 (a), the powder particles of Zirconia were effectively covered with PS and LDPE binders. The green Zirconia micro-sized component is depicted in Figure 5 (b). Injection moulded zirconia micro-sized green parts without any defects were produced using the following parameters: 10 bar of injection pressure, 70 °C of mould temperature, 190 °C of melt temperature and 7 seconds of injection time, respectively. The injection moulding parameters are reproducible and repeatable as 15 green

parts were successfully produced to undergo debinding and sintering process. It is found that temperatures less than 190 °C of melt temperature resulted in short shot defect, whereby the melted powder particles do not fill the mould fully.

CONCLUSION

The CPVP of zirconia achieved for this study was 49.92 vol.% in which the optimal loadings of 46 vol.%, 47 vol.% and 48 vol.% feedstocks were analysed using the rheometer. The results show that 46 vol.% feedstock was most suitable for producing zirconia micro-part without any defects compared to other powder loadings due to its low viscosity, high shear thinning, flow behaviour index of less than 1 and low activation energy. Therefore, 46 vol.% feedstock is the most suitable powder loading to be used in producing a defect-free zirconia micro-part through micro-powder injection moulding μ PIM process. Injection moulded zirconia micro-sized green parts without any defects were produced using the following parameters: 10 bar of injection pressure, 70 °C of mould temperature, 190 °C of melt temperature and 7 seconds of injection time, respectively. The injection moulding parameters are reproducible, experimentally accurate and repeatable as 15 green parts was successfully produced to undergo debinding and sintering process. It is found that at temperatures less than 190 °C of melt temperature resulted in short shot defect, whereby the melted powder particles does not fill the mould fully. The results showed that by employing feedstocks containing 46 vol.% of zirconia, micro-sized zirconia parts without any defects can be produced successfully through the micro-powder injection moulding process with the aforementioned optimized parameters.

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DECLARATION OF COMPETING INTEREST

None.

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