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Moldability Characteristics of 3 mol% Yttria Stabilized Zirconia Feedstock for Micro-powder Injection Molding Process

(Ciri Kebolehacuanan Bahan Suapan 3 mol% Yttria Zirconia Terstabil bagi Proses Pengacuanan Suntikan Mikro Serbuk)

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ABSTRACT

Micro-powder injection molding (μPIM) is a promising process that may satisfy the demand on miniaturization parts to micro domain in mass production with low manufacturing cost. Three mol% yttria stabilized zirconia (YSZ) with nano-sized powder and binder system consists of polyethylene glycol (PEG), polymethyl methacrylate (PMMA) and stearic acid (SA) were used. Nano-size powders with higher surface area generally require more binder to form a feedstock. As such, determination of the optimum powder loading of the feedstock for μPIM process is important. The rheological characteristics of different YSZ feedstocks with powder loading of 52 53 and 54 vol.% were investigated in terms of flow behavior as a function of viscosity and shear rate. Fairly low values of flow behavior exponent ranging from 0.25 to 0.39 (n<1) resulted in pseudoplastic flow behavior of the examined YSZ feedstock. The 52 vol.% feedstock exhibited the lowest viscosity resulting in highest activation energy and lowest moldability index of 1.862×10^{-6}, while the 54 vol.% feedstock regardless to its high viscosity, yielded a low activation energy of 4.14 kJ/mol and high moldability index of 4.59×10^{-6}. Based on rheological properties obtained, a powder loading of 54 vol.% has desirable feedstock characteristics for μPIM process and exhibited molding ability for micro detail filling. The relationship between the optimum rheological properties obtained and the actual injection process was also determined. The results showed that the green parts were able to be injected without defects such as short shot or flashing.

Keywords: Feedstock; micro-powder injection molding; rheological properties

ABSTRAK

Permintaan ke arah pengecilan komponen kecil kepada mikro saiz dalam bentuk pengeluaran secara besar-besaran dengan kos pemprosesan yang rendah telah membuatkan pengacuanan suntikan mikro serbuk merupakan satu proses yang sesuai digunakan. Bahan yang digunakan dalam proses ini ialah serbuk 3 mol% yttria zirconia terstabil (YSZ) bersaiz nano dan sistem bahan pengikat yang terdiri daripada polietilena glikol (PEG), polimetil metakrilat (PMMA) dan asid sterik. Walau bagaimanapun, serbuk bersaiz nano menyumbang ke arah luas permukaan yang lebih tinggi dengan lebih banyak bahan pengikat diperlukan, oleh itu, ia menyebabkan bahan suapan yang terhasil mempunyai pembebanan serbuk yang lebih rendah. Ini menunjukkan betapa pentingnya untuk menentukan pembebanan serbuk bagi bahan suapan untuk proses pengacuanan suntikan mikro serbuk. Ciri reologi bagi pembebanan serbuk yang berbeza iaitu 52, 53 dan 54 vol. % bagi bahan suapan YSZ dikaji daripada aspek sifat aliran dalam fungsi kelikatan dan kadar ricih. Nilai sifat aliran yang terhasil daripada kajian ini adalah agak rendah iaitu dalam julat 0.25~0.39 (n<1) menunjukkan bahan suapan YSZ yang dikaji mempunyai sifat aliran pseudoplastik diperhatikan. Bahan suapan 52 vol.% seperti yang telah dijangkakan telah mempamerkan kelikatan yang paling rendah manakala bagi bahan suapan 54 vol.%, berbanding dengan kelikatannya yang tinggi, telah menghasilkan tenaga pengaktifan yang paling rendah iaitu 4.14 kJ/mol dan nilai indeks kebolehacuanan yang tinggi iaitu 4.59×10^{-6}. Berdasarkan kepada sifat reologi ini, boleh disimpulkan bahawa bahan suapan pada pembebanan serbuk 54 vol.% memiliki ciri bahan suapan yang dikehendaki dalam proses pengacuanan suntikan mikro serbuk. Ia juga menunjukkan keupayaan diacuankan bagi mengisi penuh perincian acuan mikro. Penghubungkaitan sifat reologi kepada proses suntikan sebenar telah dibuktikan dengan jasad hijau telah berjaya disuntikkan tanpa mengalami sebarang kecacatan seperti tembakan pendek atau percitan.

Kata kunci: Bahan suapan; pengacuanan suntikan mikro serbuk; sifat reologi

INTRODUCTION

Micro powder injection molding (μPIM) is highly in demand because it is an economical process for the mass production of complex micro metal and ceramic parts (Zauner 2006). It involves the mixing of powder with a binder system, the injection of the mixture into the mold cavity, the removal of the binder system from the molded part and the sintering the part towards densification final part (German & Bose 1997). Nowadays, this process has managed to penetrate into the medical and dental fields (German 2010). Ceramic powder mostly being used to produce micro parts because it is readily available in nano-sized powder form and it is safely to use since it is not pyrophoric as metallic materials (Zauner 2006). In a report by German (2010), most of the attempt concerning μPIM medical and dental devices are focused on alumina, zirconia and stainless steel. Furthermore, the nano-powder of yttria stabilized zirconia (YSZ) has become an alternative material for medical applications due to its biocompatibility and high-strength (Piconi & Maccauro 1999). YSZ with tetragonal polycrystals structure is characterized by its outstanding mechanical properties, particularly its high bending strength, fracture toughness and Young's modulus which comparable with steel (Rieger et al. 2008). For μPIM process, nano-sized particles embedded in the matrix of YSZ feedstock facilitate the injection molding for micro- or micro-featured components. However, powders with nano-sized particles tend to agglomerate and exhibit high surface areas, resulting in poor distribution in the binder system and low solid content of the powder-binder mixture (Song & Evans 1995; With & Witbreuk 1993). Non-uniformly blended feedstock exhibits unsuitable rheological behaviors and can cause defects during molding and sintering steps, thereby resulting in poor physical and mechanical properties (Roetenberg et al. 1992). Meanwhile low powder content results in longer debinding time to remove the excessive binder and high shrinkage during sintering. A success of μPIM process is obtained when the feedstock exhibits good flow behavior characteristics. In other words, it is desirable to have low viscosity feedstock where it eases up the filling process of micro geometrics in injection molding process (Liu et al. 2001, 2002). Therefore, selection of the composition of powders during feedstock preparation in μPIM process is essential to the entire process, particularly when using nano-sized powder. Meanwhile, the rheological properties are features of the feedstock used to describe the flow behavior of the molten during the injection molding process. The present work was performed to measure the stability of the feedstock during molding by mean of the rheological analysis and to determine the optimum powder loading for YSZ feedstock. Flow behavior feedstock in a function of shear rate dependence of the viscosity can be described by (1) (German & Bose 1997):

$$\eta = K\dot{\gamma}^{n-1}, \tag{1}$$

where η is the viscosity at shear rate of $\dot{\gamma}$, K is a constant and n is a flow behavior index. It is known as the power-law equation and has been used extensively to explain the correlation of the viscosity and shear rate. Meanwhile, the relationship of temperature, shear rate and solid loading can be simplified and expressed as the moldability index as (2) (Agote et al. 2001):

$$\alpha_{stv} = \frac{1}{\eta_o}\frac{|n-1|}{\frac{E}{R}} \tag{2}$$

where η_o is a reference viscosity, E is the activation energy and R is the universal gas constant. This α_{stv} value is used to evaluate the flow behavior of the feedstock in the injection machine.

Experimental Details

Feedstock was prepared using YSZ powders (Nabond Technologies, China) containing 3% mol yttria for stabilization of the tetragonal phase. It was supplied in the form of spherical spray-dried with a mean particle size of 50 nm and specific surface area of (17±3) m^2/g. The true pycnometer density of YSZ powder was 6.4339 g/cm^3.The scanning electron microscope (SEM) images of YSZ feedstock is as shown in Figure 1. A roller blade-type Brabender mixer (GmbH & Co. KG) was used to determine the critical powder loading (CPVP) of YSZ with addition of oleic acid. Feedstock formulations containing YSZ powder loadings 2 to 5 vol% less than the critical value were prepared. The components of the binder system were PEG:PMMA:SA by ratio of 73:25:2, respectively. The binder ratio was based on previous works in which similar binder system was used (Ibrahim et al. 2009). The feedstock was blended in the Brabender mixer with a rotation speed of 25 rpm at 60°C for 90 min or until a homogeneous mixture were obtained that is, when the mixing torque (Nm) became constant. The dough like mixture was then crushed to form granulated feedstock. The rheological properties of the YSZ feedstock were measured using a Shimadzu Flowtester CFT-500D capillary rheometer. Experiments were conducted with load varying from 50~170 kgf at constant capillary temperature ranging from 140 to 160°C. The effect of powder loading on viscosity was investigated. Rheological properties were used to draft the injectability range of the critical parameters in injection process, including pressure, temperature, mold temperature and time. These parameters were used in an actual injection molding process to produce green part with no defect.

RESULTS AND DISCUSSION

Powder loading greatly influenced the rheological properties of feedstock when dealing with nano-size powder. The shape of YSZ powder generally reported is nearly spherical but it tends to agglomerate and form clusters due to van der Waals forces (Subbanna et al. 1998). A CPVP of 56 vol.% YSZ powder was obtained by adding 18 mL oleic acid into 150 g YSZ powder (Figure 2).

In this case, the optimal solid loading (2 to 5 vol.%) was relatively lower than the critical loading (51 to 54 vol.%), higher than in previous studies (Foudzi et al. 2011; Yu et al. 2009), where the critical powder loading for 3 mol% YSZ 50 nm in size was reported to be 41 vol.% solid loading. Ceramic sintered parts at lower powder loading, usually encountered in high shrinkage, high porosity and poor mechanical properties of sintered parts. According to German and Bose (1997), many ceramics are injection molded at a powder loading ranging between 50 and 55

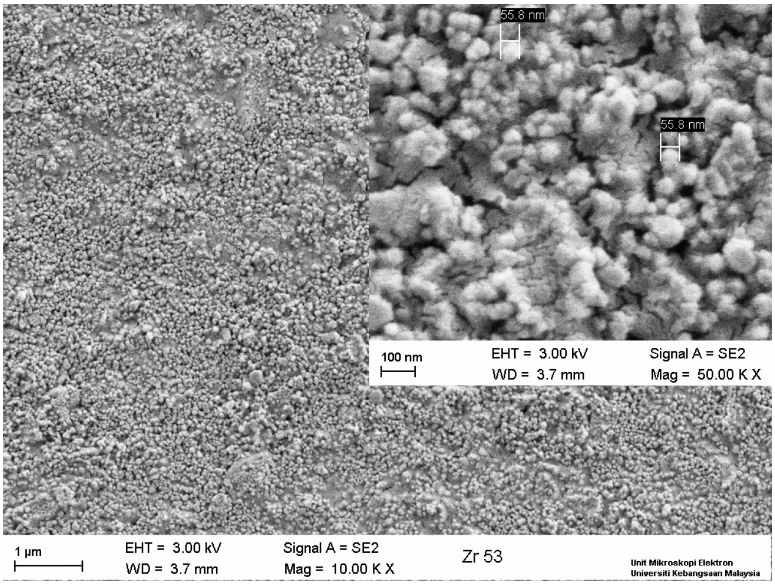

FIGURE 1. Morphology of YSZ feedstock

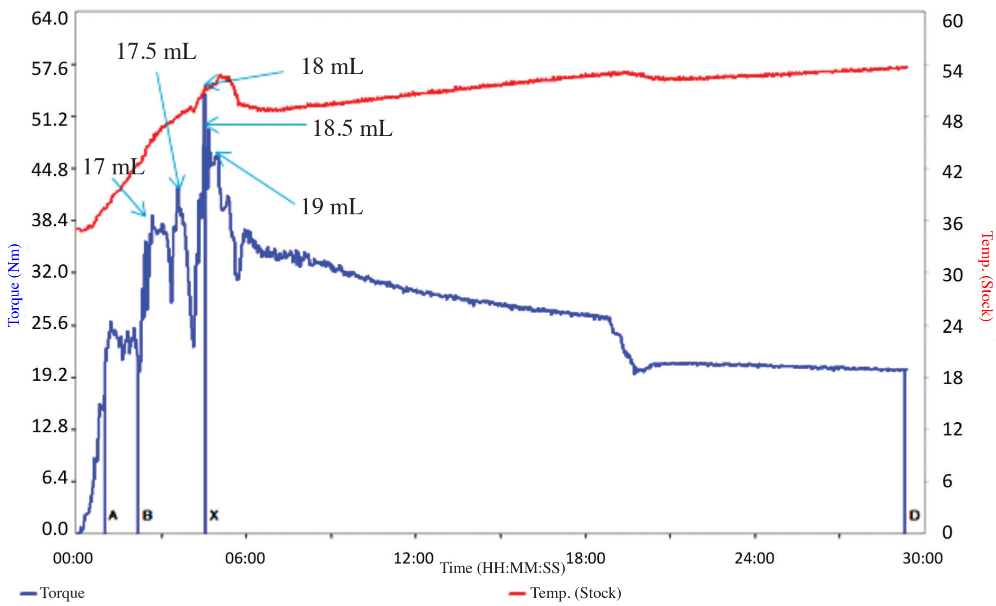

FIGURE 2. CPVP with critical peak value at 56 vol.%

vol.%. Therefore, YSZ feedstock with a loading ranging from 51 to 54 vol.% could be considered to feature optimal powder loading. A high powder loading range in a feedstock (52 to 54 vol.%) can be successfully obtained with SA in the binder formulation which acts as a lubricant or wetting agent. SA effectiveness improves as the particle surface area increases, thereby enhancing powder loading and green strength (German & Bose 1997; Omar et al. 2001; Tseng et al. 1999) and resulting in a successful μPIM process.

In mixing process, the increase in temperature and torque (Figure 3) is evidently influenced by the breaking of agglomerates in the feedstock (Khakbiz et al. 2005; Maca et al. 2002). High torque and temperature produced enough shearing stress to break agglomeration network and induced particles to align along the shear field, thereby producing a homogeneous mixture.

The scatter plots in Figure 4 demonstrates the relationship between viscosity and shear rate at 140, 150 and 160°C. The plots decay exponentially, which agrees with the power-law shown in (1). Viscosities were significantly influenced by the shear rates and depended minimally on powder loading and temperature. The viscosities decreased toward a minimum value as the shear rate increased regardless of powder loading, demonstrating the shear thinning effect. In other words, the feedstock demonstrated pseudoplastic flow, which occurs when the viscosity of the feedstock decreases with shear rate.

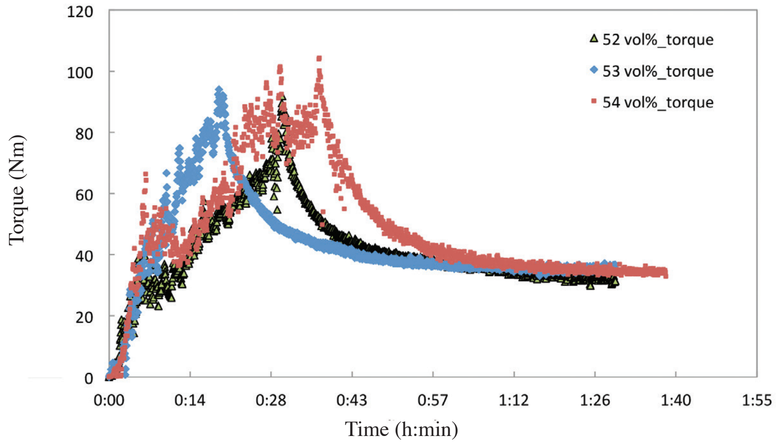

FIGURE 3. Mixing graph of YSZ feedstock with its torque stabilized at the end of the process

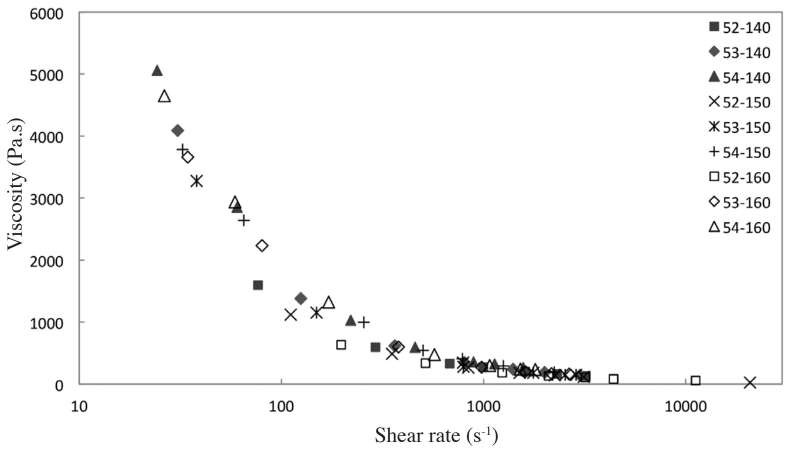

FIGURE 4. Relationship of viscosity and shear rate at different temperatures

This type of behavior is desirable for μPIM process. No powder binder separation occurred in the mixture since no dilatant flow behavior was observed. Furthermore, lower powder loading with higher SA content resulted in increased breakage of powder agglomerates to smaller fragments due to weakening of agglomeration network forces of particles, causing the feedstock to become more homogeneous and less viscous. Thus, the melted feedstock of 52 vol.% demonstrated lowest viscosity at all shear rates and temperatures which exhibited better flowability during the micro filling of complex parts in the injection molding process. In comparison, the 53 and 54 vol.% feedstock also exhibited low values of viscosity, which is less than 1000 Pa.s and within the desirable range for injection molding. Table 1 shows the viscosities of different YSZ feedstocks at different tested temperatures and shear rates. The shear rate in ceramic injection molding varies from 100 s^{-1} to 1000 s^{-1}; the flow rate during the injection molding requires a viscosity of less than 1000 Pa.s (German & Bose 1997). The viscosity in Table 1 shows that the μPIM process proceeded best at shear rate 1000 s^{-1} for all three YSZ feedstock at working temperature. From the viscosity point of view, all YSZ feedstocks could be injectable and give better flowability during micro filling of complex parts in injection molding process.

The flow behavior index, n also indicates the sensitivity of the feedstock to the shear stress, giving the insight of rheological characteristics of μPIM feedstock (Khakbiz et al. 2005). Generally, the melted feedstock is considered to be pseudoplastic fluids if n is smaller than unity (n<1), at $n > 1$, the flow is dilatant. Lower value of n signifies that flow behavior of feedstock exhibits more pseudo plasticity and sensitivity of viscosity to shear (Jamaludin et al. 2011). Defects such as cracks and voids normally occur for a high sensitivity of feedstock to shear (small n) during the injection molding (Jamaludin et al. 2011; Maca et al. 2002). Figure 5 shows the flow behavior index of the examined YSZ feedstock at different temperature derived from the exponent n of the power-law index of η-γ graphs. The values of n for all examined YSZ feedstock calculated at 1000 s^{-1} shear rate are fairly low which varied from 0.25 to 0.39 (n<1) depending on the test temperatures. The feedstock containing 54 vol.% YSZ demonstrated a slightly higher sensitivity to shear than the other feedstocks,

TABLE 1. Viscosities distribution of YSZ feedstock at different temperature and shear rate

Solid loading %	Tested temperature (ºC)	Shear rate (s^{-1})		
		240	500	1000
52	140	704.83	432.31	272.46
	150	671.13	393.90	238.14
	160	523.22	332.43	216.60
53	140	871.46	508.49	305.72
	150	811.34	473.75	285.03
	160	876.93	503.85	298.55
54	140	982.09	576.83	348.98
	150	963.41	566.28	342.83
	160	966.67	560.74	335.27

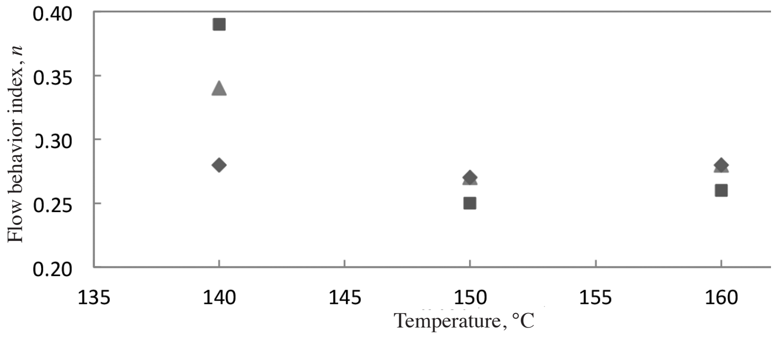

FIGURE 5. Flow behavior index of examined YSZ feedstock at different temperatures

whereas the 52 and 53 vol.% YSZ feedstocks were equally sensitive to shear as the temperature increased to 150 and 160ºC. This sensitivity could be due to the higher binder content in those feedstocks compared with the 54 vol% feedstock. From this shear rate sensitivity point of view, the 54 vol.% of YSZ feedstock evidently demonstrated the best feedstock.

In general, temperature plays a significant role in injection molding. It is known that the viscosity varies exponentially with absolute temperature *T* base on Arrhenius equation (Agote et al. 2001; German & Bose 1997; Jamaludin et al. 2011; Khakbiz et al. 2005). Figure 6 shows the correlation of viscosity and temperature in feedstock with different level of powder loading. Clearly, the viscosity of the 54 vol.% YSZ feedstock demonstrated the smallest reduction as the temperature increased. The viscosity gradients decreased as the powder loading decreased to 53 and 52 vol.% with temperature, which may be attributed to the YSZ particles that has lower thermal expansion coefficient than binder. The binder experiences greater expansion in the feedstock compared to the powder when heated and the molecular chains break as more heat is distributed to the system reducing the viscosity (German & Bose 1997; Li et al. 2007). Thus, the feedstock containing more binder (52 vol.%) experienced greatest expansion resulting lowest viscosity. Similar observation had been reported for alumina injection feedstock and also stainless steel injection feedstock (Krauss et al. 2005; Li et al. 2007). Increasing the powder loading of feedstock causes a decrease in the sensitivity of the viscosity to temperature, which helps maintaining the viscosity of the feedstock as the temperature fluctuates throughout the injection process and improves the filling efficiency in micro details parts.

The activation energy, *E* is another significant parameter used to evaluate the moldability of the μPIM feedstock. Values of *E* reflect the dependence of the feedstock on the thermal fluctuations that occur during the injection process between the injector nozzles and the mold cavity. High values of the *E* may lead to premature freezing before the melt reaches the end point of the mold cavity (Jamaludin et al. 2010). Previous studies have demonstrated that *E* may be used to identify the optimal powder loading, where the distribution values follow a parabolic trend (Contreras et al. 2010). The optimal powder loading from such trend has been determined to be approximately equal to the lowest value of *E*. In Figure 7, the distribution values of *E* decreased with powder loading following the parabolic trend. The remarkable 59% reduction of *E* at 54 vol.% feedstock indicated that such a loading is closed to the optimal powder loading. Furthermore, the low value of *E* signified the low dependence of on temperature, which is presumably attributed to lower thermal conductivity of YSZ powder (~2 W/m.K), allowing the powder to hold/store the heat for a longer time (Piconi & Maccauro 1999) such that the feedstock viscosity does not experience sudden changes when the temperature varies during molding.

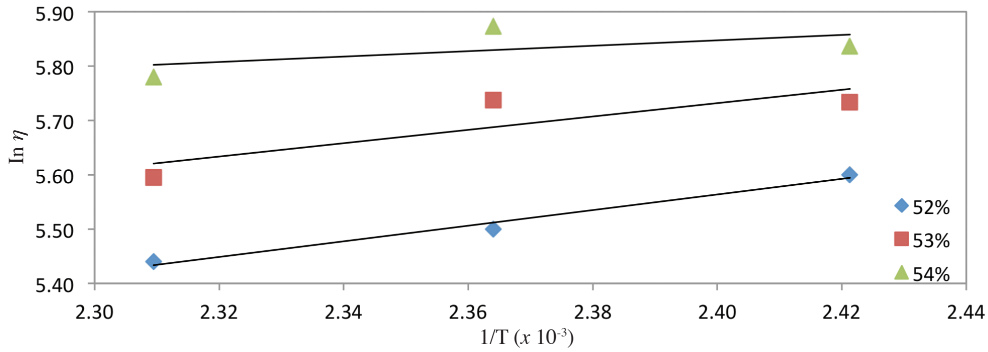

FIGURE 6. Correlation of viscosity and temperature for feedstock with different powder loading

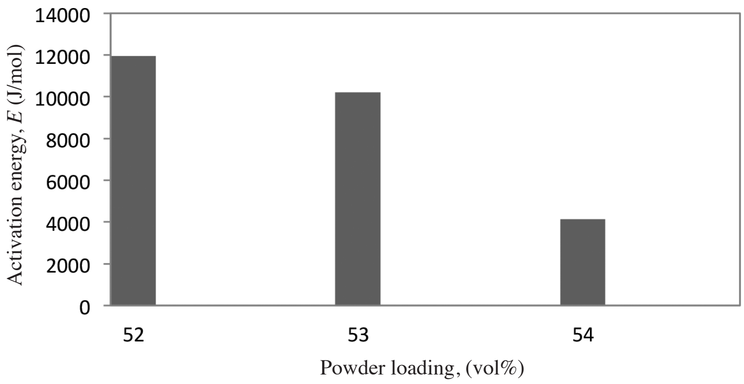

FIGURE 7. Correlation of activation energy and powder loading of YSZ feedstock

This property favors the filling of complex geometry parts. However, sudden changes in viscosity could cause undue stress concentrations in molded parts, resulting in cracking and distortion of the molded parts (German & Bose 1997; Khakbiz et al. 2005; Li et al. 2007). Thus, the 54 vol.% feedstock gives the best value as regards temperature sensitivity.

The higher the value of moldability index, α_{stv}, associated with lower value of n and E as in (2) (Weir et al. 1963) indicate optimum feedstock rheological properties. A comparison of the moldability indexes at a fixed shear rate of 1000 s^{-1} is shown in Table 2. It is observed that values of α_{stv} increases with powder loading. The feedstock with 54 vol.% powder loading exhibited a significant increase in α_{stv} at an average value of 4.242×10^{-6} as a result of significant reduction in E. Other than that, the moldability index of 52 and 53 vol.% feedstocks were fairly low and increased slightly (9%) as powder loading increased. It is noted that the feedstock with 54 vol.% powder loading exhibited the best rheological properties compared with feedstock containing 52 and 53 vol.% loading in terms of moldability. Higher powder loading is desirable during the sintering stage to decrease the shrinkage percentage and promote grain boundaries diffusion (German & Bose 1997).

The actual injection molding process was carried out at minimum values (Table 3) of the injection parameters to show the significant of rheological obtained. The injected

TABLE 2. Comparison of α_{stv} at 1000 Pa.s shear rate

Powder loading (%)	Temperature (°C)	Rheological index, α_{stv}
52	140	
	150	1.862×10^{-6}
	160	
53	140	
	150	2.036×10^{-6}
	160	
54	140	
	150	4.292×10^{-6}
	160	

TABLE 3. Minimum values of injection molding parameters

YSZ powder loading (vol%)	Injection pressure (bar)	Injection temperature (°C)	Mold temperature (°C)	Injection time (s)
52	6	145	50	5
53	7	155	60	5
54	8	160	65	6

green part (~0.05 g) appeared as micro dumb bell shape (Ibrahim et al. 2010) with dimension of 0.80 mm thickness, 3.80 mm length and 1.10 mm width. The green parts could be injected at temperature range of 145 to 160°C based on the rheology test. The feedstock with 54 vol.% powder loading required a slightly higher injection pressure compared with other feedstocks due to high shear among particles contributed at higher powder loading.

All of the injected samples have a good appearance whereby no sink mark and weld lines observed (Figure 8). Nevertheless, all feedstock could be injection molded for micro components from molding point of view. Thus, it was confirmed the relevancies of the rheological properties and the injection molding behavior.

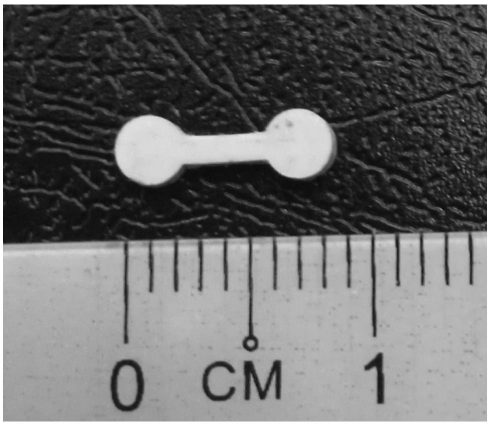

FIGURE 8. Micro dumbbell injected part

CONCLUSION

In this study, powder characteristic in the function of viscosity has significantly influenced the rheological properties of 3 mol% YSZ feedstock. The critical powder loading has greatly influenced by the particle size. The smaller the particle size, the higher the surface area thus resulted in lower powder loading. The binder system consisting of polymer promoted homogeneous mixture of high powder loading of 52, 53 and 54 vol.% feedstock and enhanced the feedstock flowability and powder packing in the presence of SA. The feedstock demonstrated a pseudoplastic flow, which favors μPIM process. Furthermore, the feedstock possessed low value of flow behavior index *n* (0.25 to 0.39), signified the sensitivity to the shear during injection process. Compared with other feedstock, the 54 vol.% YSZ feedstock exhibited the lowest *E* of 4.14 kJ/mol, thereby resulting in high average value of moldability index of 4.292×10^{-6}. The YSZ feedstock with 54 vol.% powder loading inherited the desired characteristics for μPIM process and demonstrated injectability for micro details filling. Future studies on the optimization of the injection process for the 54 vol.% YSZ feedstock are recommended.

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