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The Study of Sintering Behavior of Stainless Steel 17-4PH-Stainless Steel 316L for Two Materials Powders Injection Molding (2C-PIM) Process

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ABSTRACT

Two materials Powder Injection Molding (2C-PIM) is a recently developed method to manufacture functionally graded components. This study is focused on determining the suitability of two materials combined via experimental PIM technique by the sintering and microstructural evaluations. In addition, the shrinkage behavior between the two materials is also observed. The materials in 2C-PIM are said to be compatible with each other in terms of metallurgical bonding and responsive during the sintering process. The materials are stainless steels 17-4PH and 316L with palm stearin and polyethylene as the binders. It has been found that the difference sintering shrinkage for both materials is not significant due to the coefficient thermal expansion (CTE) between the two materials are quite similar. Therefore stainless steels 17-4PH and 316L can be combined successfully via the 2C-PIM technique.

Keywords: *co-powder injection molding; dilatometer; rheology; sintering; stainless steel*

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Introduction

Powder injection molding (PIM) is a powder metallurgy net shaping process that allows complex parts to be mass manufactured with metal, ceramic, or composite materials [1]. Such process consists of four main steps: mixing, injection molding, debinding, and sintering. These four steps are also practice in 2C-PIM or also can be called as Co-PIM. However, the difference between PIM and 2C-PIM is during the injection molding step where 2C-PIM may be implemented in two ways either over-molding or co-injection molding [2]. In the over-molding, two different barrels are used to inject two different materials into a desired shape while co-injection molding is a functionally graded structure which produces the desired part using the flow behavior of materials through the same runner system while co-injection molding, can produce a component that has a core and skin made of two different materials [2].

According to the previous research, additional process such as secondary process will spend more cost compared to by using 2C-PIM process [3]. The secondary process is referred to produce interlayer or any combination of two materials. Therefore, by using one of the 2C-PIM method, it will reduce the production cost. Apart from can reduce the extra manufacturing process, sintering process in 2C-PIM also is one of the improvements over the welding proses or similar approaches involving adherence to each other [4]. From previous research, the co-sintering of alumina and zirconia in tape-cast green sheets has been studied [5]. It was found that such sintering resulted to various defects at the intermediate. For example, channel cracking due to tension and delamination which is due compression and debonding. These defects attributed to the mismatch of thermal expansion for both materials. Therefore, shrinkage behavior is a vital factor to implement the 2C-PIM process efficiently.

In this paper, dilatometry is used as an experiment method to determine the compatibility of two different materials for two metal powder injection molding. The co-sintering behavior and shrinkage behavior can identified separately through this method. Both behavior that has been selected by materials. The objective of this experimental study was to assess suitable material systems for co-sintering process in powder injection molding. Not only can examine the most similar thermal shrinkage rate, the dilatometer study also was selected because of their capability to give favorable interfacial integrity after injection molding and co-sintering [2].

There are also challenges relating to the inherent powder characteristics such as porosity or contamination which affect structural properties of a joint and performance or integrity. The powder characteristics which include particle shape, size and surface area influence porosity which consequently affect the physical properties. The problem is how to obtain a sound bonding between the dissimilar materials. Though some impressive results have been recorded, successful implementation of this technology remains summarily an ambitious challenge. Thus 2C-PIM process has continued to be a subject of intensive research. However, investigations reveal that direct joining of zirconia to metals is feasible if appropriate processing condition is employed [6].

The concept of 2C-PIM is based on joining of the ceramic-metal composite in their green states and subsequently, obtaining the desired density through further processing which includes debinding and sintering. So far, various processing techniques utilizing interlayer material for joining have been established [7,8]. In these techniques, the disadvantage of inclusion of a third material different from the base materials and additional manufacturing step are usually imposed [9]. Thus, direct joining of composites such as through 2C-PIM process ensures reduction in the process chain and complexity in addition to assembly cost savings.

Methodology

The two stainless steels; 17-4PH and 316L are mixed separately with a binder system consists of 60wt% palm stearin (PS) and 40wt% polyethylene (PE). Palm stearin helps in decreasing the viscosity of feedstock and increase the replication ability while PS is functioned as modifier to reduce the viscosity and yield stress of the mixture. Prior to mixing, the critical powder volume percentage (CPVP) analysis was conducted to determine the optimum powder loading for the feedstock using the Brabender mixer. Such optimum powder loading leads to feedstock stability that prevents the powder-binder separation. Each stainless steel is then mixed with the binder system separately at 150 ºC for 1 hour using the same mixer.

The injection molding process is then conducted using the BOY Machine 22A. The co-injection molding variants are utilized in order to conduct the 2C-PIM technique. Stainless steel 17-4PH feedstock is injected first followed by SS316L feedstock using the same barrel. For initial experimental work, the green part for each materials is produced first due to find the relevant parameters which can be used for 2C-PIM process. Table 1 shows the injection molding parameters to prepare the feedstocks in order to control the possibility of defects formations at the interface. The same parameters is used for producing the 2C-PIM green part.

After producing the green part, the solvent debinding process was performed in a vacuum oven (IND#NSI). Heptane was used as a solution in terms of removing the PS binder. Temperature was maintain at 60 °C for 5 hours in the vacuum oven. Then, the tube furnace is needed in order to conduct the critical process in injection molding which is co-sintering process. After many trials is done by using several types of furnace, only tube furnace can be succeeded to implement the co-sintering process for this study. Therefore, the temperature that used in this process is 1200°C for 3 hours.

Table 1: Injection Molding Parameters

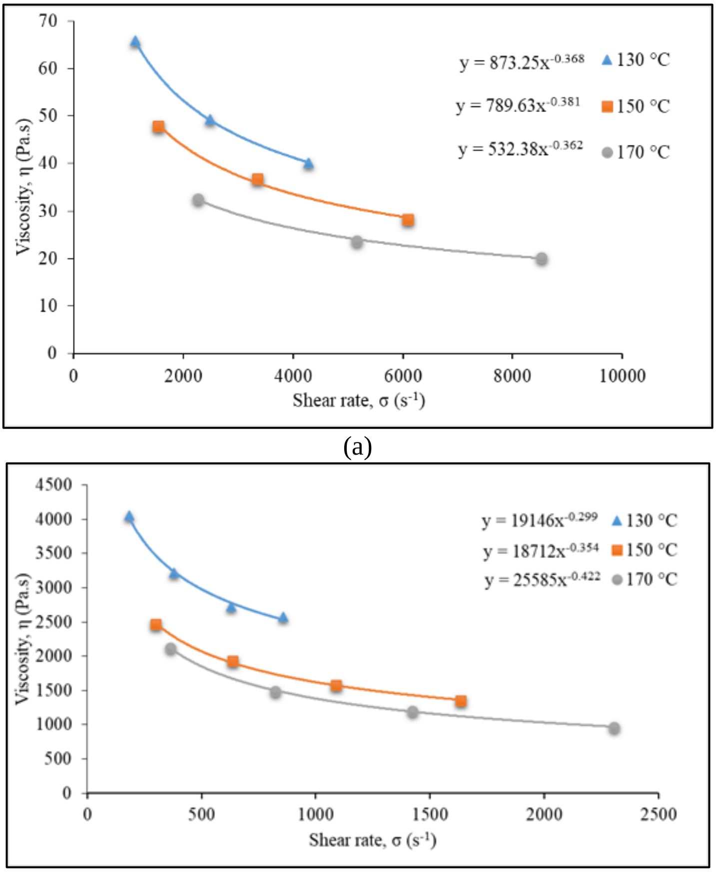
Mold Temp. (°C)	Injection Temperature (°C)					Holding Pressure (bar)	
	T1	T2	T3	T4	T5	P1	P2
35	175	165	165	165	150	145	150

However, before proceed with sintering process, the sintering shrinkage of the injected 2C-PIM is also compared with that of the injected single material produced by the same injection molding machine. Such comparison is needed for better evaluation regarding the thermal expansion mismatch due to two different materials. The debound part is then presintered using a dilatometer at 1200 °C and 5 °C/min in order to identify the shrinkage behavior for both materials. The dilatometer study is conducted by using vertical dilatometer Linseis Model for each material. In order to verify the dilatometer results, three sets of trials have been approached. After all, the micrograph is observed through the SEM image.

Results and Discussions

The feedstocks are granulated and investigated for their rheological properties using a capillary rheometer. Figure 1 (a) and (b) show the shear rate-viscosity curves for the SS316L and SS17-4PH feedstocks at three different temperatures, respectively.

The preferable PIM feedstock possess pseudoplastic flow that decrease in viscosity and increase in shear rate. Figure 1 (a) and (b) show the viscosity-shear rate curves for 72vol% SS17-4PH and 64vol% SS316L feedstocks, respectively, at three different temperatures 130°C, 150°C and 170°C. Both feedstocks show pseudoplastic flow behavior where the viscosity is less than 1000 Pa.s and the shear rate is between 102 to 105 s-1. Such behavior eases the mold filling, minimizes jetting and helps to maintain the shape of the molded part [10]. In addition, the temperature and pressure during injection molding can also be reduced.



(a)

(b)

Figure 1: (a) Shear rate viscosity of SS316L and (b) Shear rate viscosity of SS17-4PH.

Molding is a critical stage that replicates the shape of the mold cavity on the PIM material. Many associated problems at this stage arised due to powder-binder incompatibility [11]. It is therefore important that the optimal binder content is precisely estimated based on the powder characteristics to ensure that binder envelopes the powder particle completely by a thin film. The amount of binder is related to particle packing. Thus, molding must be performed at a solid content with slightly more binder than that corresponding to the critical solid loading [12]. Excessive binder causes powder-binder separation and in-homogeneities in molded parts while insufficient binder leads to voids or trapped air pockets which result to high viscosity molding.

The molding parameters are heavily interactive at any particular molding condition, thus the solution to one problem can lead to a defect in another form. The critical parameters include; injection temperature, mold temperature, injection speed, injection pressure and cooling time. The injection temperature must typically be set above the melting point of the higher molecular binder or backbone binder. This temperature influences the viscosity of the melt and consequently the ability to fill the mold cavity. Too low temperature setting can result in flow lines evident on the part due to poor flow characteristics [13].

On the other hand, higher temperatures can cause blister effects. The mold temperature must also be set at a temperature below the melting point and recrystallization temperature of the lower molecular weight binder component or primary binder. The mold temperature affects the development of stresses, rate of cooling and filling of the mold cavity. Too low mold temperature can lead to incomplete filling of the part or other defects while higher mold temperatures can lead to blisters and flashing [13]. The injection speed is important to ensure the die cavity promptly fills in a short time while injection pressure drives the filling of the mold. Adequate cooling time is also essential for redistribution of internal stresses and consequently, satisfactory part [14].

The co-injected part of SS17-4PH/SS316L is shown in Figure 2. It is observed that both materials can be combined via 2C-PIM using the same injection molding parameters as used by a single material. In addition, no visible defects are observed on the surface of the injected green part.

Figure 2: Green part of 2C-PIM

In this investigation, the barrel type injection molding (BOY 22A) was manipulated to process the green composite. In one sequence, feedstock of the first material was fed to produce a semi-finished part. The semi-finished part was replaced in the mold and the second of feedstock material was fed to complete the part in another sequence. Good bonding of SS17-4PH with SS316L in the green state was obtained and no flaw was observed. However, a standard equipment for sequential molding is needed by utilizes two injection units in order to inject two different feedstocks into the required shape [13].

Following the molding of one part in the cavity, the tooling is rotated to open up another cavity for molding of the remaining part which interlocks the previous part. It has been reported that a very narrow interface is usually obtained when sequential molding approach is employed compared to simultaneous molding method [14]. This is understandable due to existence of temperature gradient between the two parts since one part cools down before the second part is injected.

In addition, the shrinkage behavior during the sintering process for 2C-PIM samples was observed through dilatometer study. Based on Figure 3, it was found that the difference of shrinkage percentage between these two materials is similar. Therefore, in order to prove such finding, co-sintering process is implemented.

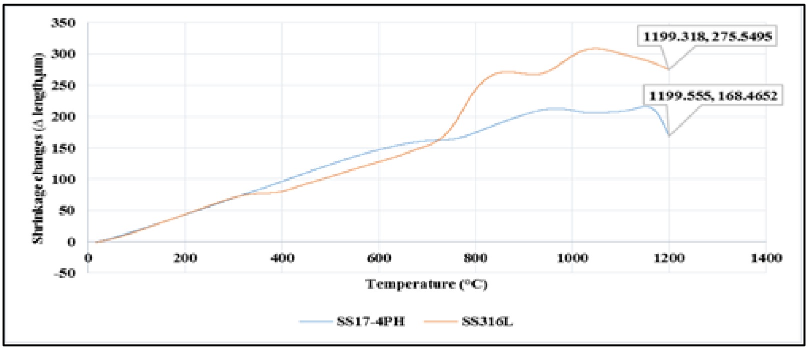
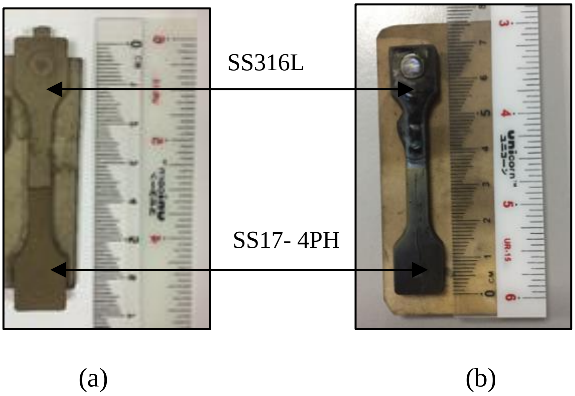


Figure 3: Shrinkage Percentage of SS17-4PH and SS316L from Dilatometer Study

The experiment work is continued in order to verify the dilatometer results. All the four main stages in 2C-PIM such as mixing, injection molding, debinding and sintering are conducted. The co-injection molding process is found to be successful, as shown by Figure 2. The injected part is then debound in two stages; solvent and thermal debinding. However, there were many challenges that occurred during the debinding process. Such challenges lead to three sets of trial that were conducted in this study.

For the first trial, debinding process is conducted in solvent and thermal. Solvent debinding is carried out first followed by thermal debinding. Solvent debinding process is conducted by immersing the co-injected parts into heptane solution at 60 °C for 4-5 hours in order to remove PS. PE is then removed by thermal debinding which is conducted at 500 °C for 1 hour in tube furnace with argon environment. However, such practice leads to poor joining where spaces between SS17-4PH and SS316L were visibly observed as shown in Figure 4(a). Therefore, it can be said debinding in two stages; solvent and thermal, is not appropriate for co-injected SS17-4PH and SS316L parts.

For the second trial, only thermal debinding is conducted using the same parameters used in the first trial. That means, solvent debind was skipped for the second trial. Unfortunately, very poor condition of debound part obtained, as shown in Figure 4(b). This may due to the lacking process of solvent debinding. Based on the PIM theory, the purpose of solvent debinding is to remove the binder where such removal creates pores structure for easing the further binder removal during the thermal debinding stage [12]. Therefore, by not having such open pore structure, the binders which is PE will be very difficult to be removed and resulted to poor surface or condition of the debound parts where excessive burnout and flashing of the binders may be observed.



(a) (b)

Figure (4): (a) Brown parts after solvent and thermal debinding, (b) Brown part after thermal debinding only

For the final trial, the debinding process is similar with the second trial except for the thermal debinding where the process was incorporated in the sintering process. Therefore, two stages of heating were implemented after the solvent debinding is completed. That means, the part will be heated at two different temperatures; 500 °C and 1250 °C in a tube furnace. The first heating at 500 °C is to remove PE while the second heating at 1250 °C is for sintering, as shown by the sintering profile in Figure 5. Based on such approach, the co-injected SS17-4PH and SS316L part is debound and sintered successfully.

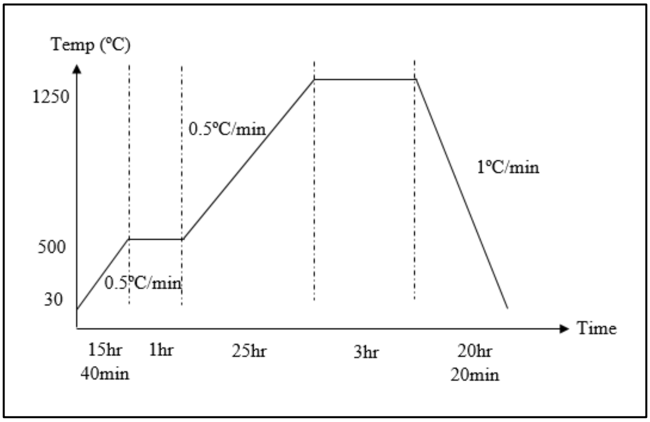
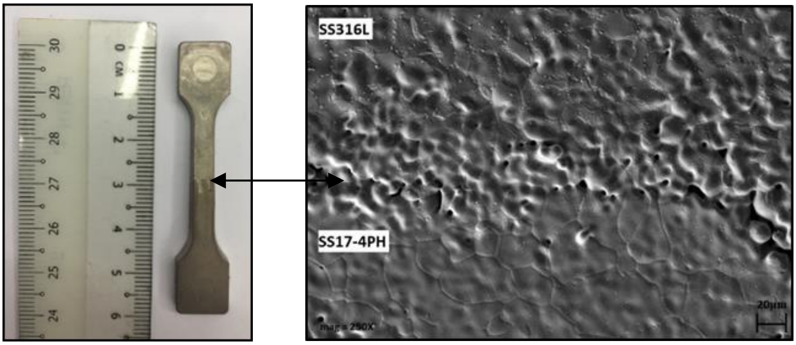


Figure 5: Sintering profile for third trial

Such co-sintered part is also bonded completely and proved by the SEM image as shown in Figure 6(b).



(a) (b)

Figure 6: (a) Captured image of 2C-PIM SS17-4PH and SS316L sintered part at 1250 °C and (b) SEM micrograph of the joining section between SS17-4PH and SS316L.

Conclusions

2C-PIM of SS17-4PH and SS316L was successfully injected where no visible defects on the surface were observed. Such success may be achieved by the binder system that showed promising rheological properties as required in PIM. In addition, the mixing was conducted well where no powder-binder separation occurred due to the homogeneous mixture of the feedstocks. The suitable powder loading for SS17-4PH and SS316L were 72vol% and 64vol%, respectively. Dilatometer study also proved that both materials can be injected in 2C-PIM or Co-PIM process because the co-sintering process was successfully implemented without any defects. Although many trials have been done, but the best method and parameters on debinding and sintering process has been succesfully evaluated in this study.

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