

## Debinding Process of Co-Powder Injection Molded (2C-PIM) SS17-4PH and SS316L

Najlaa Nazihah Mas'ood<sup>a\*</sup>, Abu Bakar Sulong<sup>a</sup>, Norhamidi Muhamad<sup>a</sup>, Farhana Mohd Foudzi<sup>a</sup>  
Intan Fadhlina Mohamed<sup>a</sup> & Al-Furjan M.S.H<sup>b</sup>

<sup>a</sup>Department of Mechanical and Manufacturing Engineering,  
Faculty of Engineering & Built Environment, Universiti Kebangsaan Malaysia, Malaysia.

<sup>b</sup>School of Mechanical Engineering,  
Hangzhou Dianzi University, Hangzhou, 310018, China.

\*Corresponding author: najlaa.elle@gmail.com

Received 08 October 2019, Received in revised form 30 June 2020

Accepted 01 July 2020, Available online 30 November 2020

### ABSTRACT

*Co-Injection Molding Process (2C-PIM) is the extension of powder injection molding (PIM) process which is widely used in manufacturing industry. However, due to two materials being injected, the development of required parts by 2C-PIM can be challenging. Yet, there are many researches that have been done by 2C-PIM process because the process is capable of combining metal-metal, ceramics-metal and ceramics-ceramics. The selections and combinations of materials are depending on the targeted applications. The 2C-PIM process has 4 main processes which are mixing, injection, debinding and sintering. In this paper, SS17-4PH and SS316L were used to conduct the 2C-PIM process. Both materials were mixed with the binder system which is 60 wt.% of palm stearin (PS) and 40 wt.% of polyethylene (PE) to produce as feedstock. The green parts of SS17-4PH and SS316L were prepared by co-injection process. The co-debinding process for these green parts was investigated. Such process was divided into two stages: solvent debinding and thermal debinding. Solvent debinding was conducted with heptane solution at 60 °C. Meanwhile, thermal debinding was conducted at 500 °C for 1 hour holding time and continued with sintering process in the same furnace at 1250 °C. It was found that the binders were 95% removed during solvent co-debinding and such finding is adequate for the co-sintering process based on PIM concept. The co-sintered part was successfully produced and the physical properties of the co-sintered part was observed in order to ensure the bonding is good. Result of SEM image has proved that the co-sintered part of SS17-4PH/SS316L is well bonded.*

*Keywords: Co-injection molding; powder injection molding; solvent debinding; thermal debinding; brown parts*

### INTRODUCTION

Powder Injection Molding (PIM) is applicable to metals and ceramics. This process combines a small quantity of a polymer with an inorganic powder to form a feedstock that can be molded. Powder Injection Molding is one of the technologies for mass-producing near net shape components which can produce micro components. This technology consists of four main processing steps: i) mixing of powders and selected binders to get the feedstock, ii) injection molding of the feedstock, iii) debinding to remove the binder, and iv) sintering that occurs close to melting temperature for the material (German and Bose 1997). The main processing steps are also applicable and identical to the co-injection molding (2C-PIM or Co-PIM) process. The 2C-PIM process is only using two different types of powders as their material.

Every process has their own limitations. 2C-PIM process also has few limitations. From previous studies, the major problem of 2C-PIM process that has been reported is the different shrinkage of both materials. This problem will occur during co-sintering process because both materials

will shrink together in the same time when high temperature is applied (Oh et al. 2017; Imgrund et al. 2008). However, each material has their own shrinkage behavior. Therefore, dilatometer testing is needed to examine the percentage of shrinkage for each material in order to ensure the co-sintering process for 2C-PIM can be done successfully (Oh et al. 2017; Quinard et al. 2009). If the shrinkage difference between two materials is too big, defect will occur at the sintered part such as mismatch defect.

Binders selection is one of the important things to take into account when performing the powder injection molding process. It is because when the combination of selected binders and powders are not suitable, it will bring many issues especially binder-powder separation, powder agglomeration and poor strength of green part. All of these issues will result in distortion, cracking and blistering of parts (Ani et al. 2014). Following the binder selection step, the process of binder removal is also a very critical step due to the possibility of defects appearance. The best binder removal or debinding process will keep the green part good for further co-sintering process. The debinding process includes solvent, thermal, evaporation

and catalytic debinding (Trunee et al. 2002; Li 1998). If possible, the combination of these approaches may also be conducted.

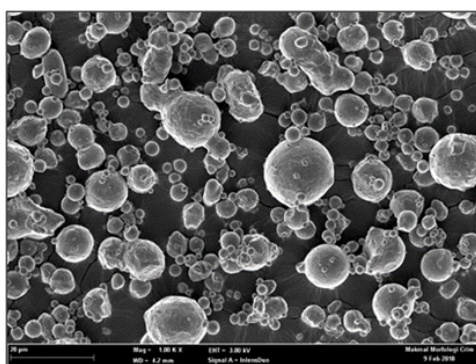
In debinding process, the binders are usually including lubricant, surfactant and backbone binder (Ani et al. 2014; Loebbecke et al. 2009; Yu et al. 2007). These three types of binders have their own functions where lubricant will act as a filler phase, surfactant will act as a bridge in binder and powder that has used and backbone binders is to keep the shape integrity. Lubricant and surfactant can be removed by immersing the green part in solvent while backbone only can be removed in thermal decomposition (Manman 2019).

Most of previous studies have reported that thermal debinding and sintering processes are done separately (Chang 2019). However, these two processes can be combined due to the limitations (Fayyaz et al. 2014). The limitation is difficulty of handling the brown part after thermal debinding process. Therefore, the combination of both processes is required by doing two-stages of heating temperature in the same furnace in order to produce the sintered part without any defects.

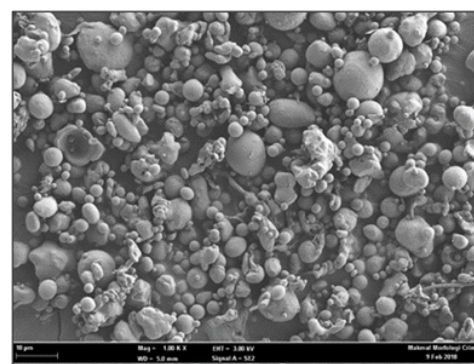
## METHODOLOGY

### EXPERIMENTAL MATERIALS

In this study, two materials have been selected which are Stainless steel 17-4PH (SS17-4PH) powder and Stainless steel 316L (SS316L) powder of average size 20 $\mu$ m and 7 $\mu$ m respectively as shown in Figure 1 (a) and (b). Both materials are used in order to conduct 2C-PIM process. Two types of binders that were used for each powder mixing are palm stearin (PS) and polyethylene (PE) with 60 wt.% and 40 wt.% respectively.



(a)



(b)

FIGURE 1. (a) SEM of SS17-4PH and (b) SEM of SS316L

TABLE 1. Injection Molding Parameters

Mold Temp. (°C)	Injection Temp. (°C)	Injection Pressure (bar)	Filling Phase (mm)	
			Single (SS17-4PH)	2C-PIM
35	150	150	24	14

Mixing process is used to produce or prepare feedstock. Preparing feedstock has been done by using Brabender mixer at 150 °C since the higher melting point of PE binder that was used is 120-150 °C. Both powders are mixed separately with powder loading of 72 vol.% and 64 vol.% respectively which is selected based on previous rheological study (Mas'ood et al. 2018). Both powder loadings are relevant and fulfilled the PIM concept for metal powders (German & Bose, 1998). In addition, previous study has applied this range of powder loading which is 65 vol.% to 70 vol.% (Sotomayor et al. 2010). The mixing process for both powders will take about 1 hour in order to ensure the feedstock is homogeneous.

After the mixing process, the injection molding process is conducted. This process has been implemented by using BOY 22A machine. In order to control the behavior of the green part for both materials, the parameters that were used to inject the first and second material are the same. In this study, SS17-4PH acts as the first material while SS316L will be the second material. It means, the first material will be injected first then followed by the second material in accordance with the 2C-PIM process. The parameters that were used as shown in Table 1.

### DEBINDING AND CO-SINTERING PROCESS

Since this study is conducted for co-injected parts, therefore, the co-debinding process must be implemented wisely. It is to ensure the brown part is still in good condition for the following co-sintering process. The two-stage co-debinding process containing solvent debinding and thermal debinding was utilized to remove the binders in the green parts. n-heptane has been used as a solution in order to conduct the solvent debinding. The green parts were immersed in

the solution at 60 °C for 4-5 hours. In order to ensure this duration is the optimum time in removing the PE binders, the percentages of binder removal were calculated. It was calculated based on the weight of the green part through the time which is an hour of solvent debinding. This step will remove the soluble binder which is PS.

However, in order to remove PE binder, there are some problems to maintain the appearance of the brown parts from any defects. Therefore, after several studies, the thermal debinding process is carried out in furnace together with co-sintering process. The furnace that was involved to carry out both processes is tube furnace. In these processes, double stages of heating are required in the furnace which is the first stage aims to remove the backbone component (PE) while the second stage is for co-sintering process. All the green parts after solvent debinding were put in the furnace with the controlling heating rate of 0.5 °C/min from room temperature to 500 °C for 1 h and then were continued fire at the same heating rate at 1250 °C for 3 h in argon as shown in Figure 2. These parameters are also based on related studies that already succeeded in 2C-PIM process (Aneta and Bogucki 2018).

## RESULTS AND DISCUSSION

### SOLVENT DEBINDING

The debinding process of the green parts was implemented in two stages: solvent and thermal debinding which have been combined with the co-sintering process. As explained before, solvent debinding has been done by immersing the co-injected parts into n-heptane for 4-5 hours at 60°C. This method was selected because PS can be dissolved through

immersion in n-heptane (Mohamad Nor et al. 2013). The duration to remove the PS binders has been recognized based on the weight reduction of the co-injected part every one hour immersed.

According to German and Bose, the waxes or oil binders must be removed more than 30% of the binder and also can be up to 98%. Therefore, in this study, the PS binder has been succeeded to remove 92% to 95% of the PS binder as shown in Figure 3(a). This experimental was conducted for 3 samples of co-injected parts in 5 hours. Although some of the previous research has been done until 6 hours of immersion in n-heptane, but in this study, at 6<sup>th</sup> hour of immersion, the co-injected parts cannot withstand anymore because it sustained damage. Figure 3(b) shows the green part after solvent debinding for 5 hours without any defects. Therefore, the time of immersion is also depending on the materials.

### THERMAL DEBINDING WITH CO-SINTERING PROCESS

In perfecting the debinding process which is removing the backbone PE binders, the thermal debinding is needed. However, through this study, the single process in removing the PE binder by using a debinding Split Furnace model RS 800/200/200 at 500°C for 4hr, was failed. Therefore, the combining process of co-sintering process has been implemented. Through this process, double stages of heating at 500°C and 1250°C with heating rate 0.5°C is required. The first temperature is needed in order to remove the PE binders while the second temperature is to fire the samples for sintering process. 1250°C was selected because of the melting temperature for both materials are about 1500°C. All these parameters as explained before has been identified

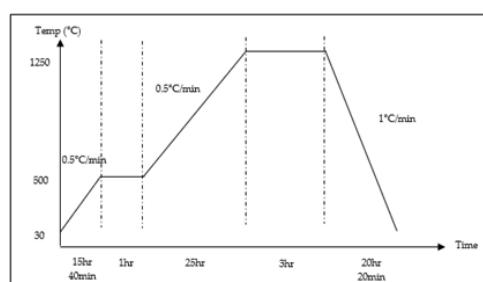
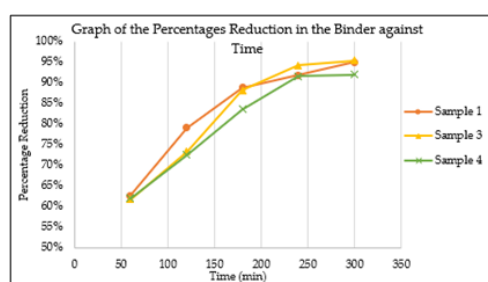
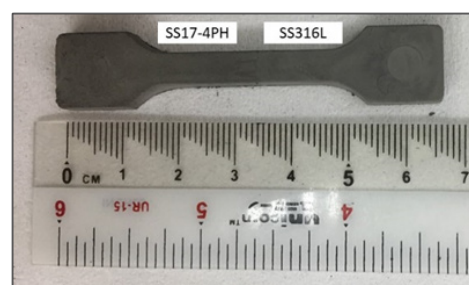


FIGURE 2. Thermal co-debinding and co-sintering phase



(a)



(b)

FIGURE 3. (a) Graph of Percentage Reduction in Binder Removal (b) Green Part After Solvent Debinding



FIGURE 4. Co-sintered part of SS316L and SS17-4PH



FIGURE 5. Failure in Co-sintering process

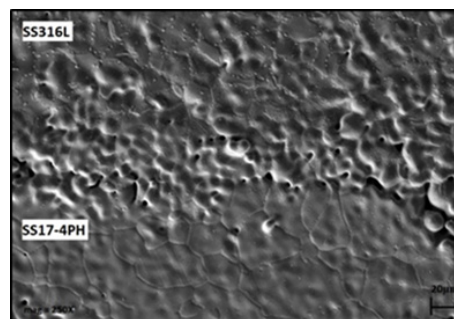


FIGURE 6. SEM Image of Co-Sintered Part

after several trial and failure process. The co-sintered part has been produced successfully as shown in Figure 4.

As discussed before, the heating rate used in this experimental is very low in order to avoid from crack occurrence to the co-sintered part. This study has also found that when heating rate of 1.0 °C/min was used during these processes, the co-sintered part cannot be produced as shown in Figure 5. It is because the shrinkage behavior has to be tackled by using very low heating rate.

However, the 2C-PIM process between SS17-4PH and SS316L has also been reported that it can be joined successfully (Aneta and Bogucki, 2018; Simchi et al. 2006). The research has been confirmed through SEM image that was conducted by researcher and it shows that both materials have bonded tightly as in Figure 6.

#### CONCLUSION

Debinding process was performed to determine the best techniques for co-injected parts without any defects. Two types of debinding processes were involved in this study. The first one is solvent debinding by immersion in n-heptane at 60°C for 5 hours. This duration was considered as the optimum time to eliminate the PS binder in the co-injected part because of the increased rate of binder weight loss. The percentage of binder removal from the co-injected part is almost 95% for all three samples and the value has been accepted in injection molding process. While the second types of debinding process is thermal debinding process which is combined with co-sintering process with two stages of heating temperature. All the binders in co-injected

parts are successfully eliminated by combining the steps in order to prevent occurrence of any defects occur in the co-sintered part.

#### ACKNOWLEDGEMENT

The authors would like to thank the Ministry of Higher Education and Universiti Kebangsaan Malaysia for their financial support under the grant FRGS/1/2013/TK04/UKM/01/2 and DIP/2016/009.

#### DECLARATION OF COMPETING INTEREST

None.

#### REFERENCES

- Aneta, S. & Bogucki, R. 2018. Sinter-bonding of AISI 316L and 17-4PH stainless steel. *Journals of Materials Engineering and Performance*.
- Ani, S.M., Muchtar, A., Muhamad, N. & Ghani, J.A. 2014. Binder removal via two stages debinding process for ceramic injection molding. *Journal Ceramics International* 4(2):2819-2824.
- Chang, W.G., Oh, J.W., Song, G.W., Shin, D.S. & Park, S.J. 2019. Rheological and thermal debinding behaviors of silicon nitride in powder injection molding. *Ceramics International*.
- Fayyaz, A., Muhamad, N., Sulong, A.B., Rajabi, J. & Wong, Y.N. 2013. Fabrication of cemented tungsten carbide components by micro-powder injection molding 214(7):1436-1444.
- German, R. & Bose, A. 1997. Injection Molding of Metals and Ceramics. *New Jersey: Princeton*.



- Imgrund, Ph., Rota, A. & Simchi, A. 2008. Microinjection molding of 316L/17-4PH and 316L/Fe powders for fabrication of magnetic-nonmagnetics bimetals. *Journal of Materials Processing Technology* 200(1):259-264.
- Li, Y. 1998. Review on debinding technique in metal injection molding. *Materials Science Engineering Powder Metallurgy* 2(1):26-32.
- Loebbecke, B., Knitter, R. & Haubelt, J. 2009. Rheological properties of alumina feedstocks for the low-pressure injection molding. *Journal of Europe Ceramics Society* 29(9): 1595-1602.
- Manman, Z., Liang, Q., Jingwu, Z., Yao, Y., Yu, Y., Wnagchang, L. & Shenglei, C. 2019. Investigation of solvent debinding in the injection molding of ZrO<sub>2</sub> ceramics using LDPE, HDPE and wax binders. *Journals of Ceramics International* 44: 3894-3901.
- Mohamad Nor, N.H., Muhamad, N, Mohd Ihsan, A.K.A. & Jamaludin, K.R. 2013. Sintering parameter optimization of Ti-6Al-4V metal injection molding for gighest strength using palm stearin binder. *Procedia Engineering* 68:359-364.
- Mas'ood, N.N., Sulong, A.B., Muhamad, N., Mohamed, I.F., Foudzi, F.M., Ramli, M.I., Salleh, F.M. 2018. Flow behaviour and injectability of SS17-4PH with PS and PE binders in powder injection molding. *Journal of Advanced Research in Fluid Mechanics and Thermal Sciences* 49(2): 121-125.
- Oh, J.W., Ryu, S.K, Lee, W.S & Park, S.J. 2017. Analysis of compaction and sintering behaviour of 316L stainless steel nano/micro bimodal powder. *Powder Technology* 322:1-8.
- Quinard, C., Barriere, T. & Gelin, J.C. 2009. Development and property identification of 316L stainless steel feedstock for PIM and  $\mu$ PIM. *Powder Technology* 190 (2):123-128.
- Simchi, A., Rota, A., & Imgrund, P. 2006. An investigation on the sintering behaviour of 316L and 17-4PH stainless steel powder for graded composites. *Materials Science and Engineering*.
- Sotomayor, M.E., Levenfeld, B. & Varez, A. 2010. Powder injection molding of premixed ferritic and austenitic stainless steel powders. *Materials Science and Engineering A* 528(9):3480-3488.
- Trunee, M. & Chiller, J. 2002. Thermal removal of multicomponent binder from ceramics injection moldings. *Journal of Europe Ceramic Society* 22(12):2231-2241.
- Yu, P.C., Li, Q.F., Fuh, J.Y.H., Li, T. & Lu, L. Two stage sintering of nano-sized yttria stabilized zirconia process by powder injection molding. *Journals of Materials Process Technology* 192(5): 312-318.