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The Spiral Flow Analysis on the Injection Molding of Alumina Powder — An Experimental Design

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Abstract: A spiral flow analysis based on the McLean–Anderson design method was conducted in studying the influence of each formulation component on the flow of alumina feedstocks. The mixtures were composed of alumina powder (AL), polypropylene resin (PP), paraffin wax (PW) and stearic acid (SA). Fourteen different formulations were designed and tested. The spiral flow distance (SFD) of each formulation as a function of ingredient concentration was measured and analyzed. The studied ranges of AL, PP, PW and SA were 87–89, 5–9·25, 3–7·25 and 0·75–1·5 wt%, respectively. It is observed that lower alumina content or higher binder content would obtain longer SFD of feedstocks. Both PW and SA are more effective than polypropylene (PP) in enhancing SFD. For alumina content less than 88%, PW is the most effective one. In contrast, SA becomes the most effective when the powder loading is higher than 88%.

INTRODUCTION

Powder injection molding (PIM) is an advanced technology that has received much attention lately for processing metal and ceramic powders. It can mass-produce good compacts with complex shape at a faster speed.[1,2]

Suitable fluidity and plasticity of feedstocks provided by binders are critical in PIM. It sustains the powder and makes a homogeneous mix free from agglomerates.[3] Most importantly, it is essential to make moldings without defects. Failure to disperse the powder uniformly or poor rheological behavior of material will cause molding defects such as distortion, cracks or voids, that will further lead to nonuniform shrinkage, blisterings or warping in the final sintered compact.[4] Consequently, both characterization and control of the rheological properties of feedstocks are necessary.

There are three approaches in characterizing the rheological properties that are relevant to molding behavior:[5] (1) measurements of viscosity in a rheometer over a range of temperature and shear rate, (2) spiral flow molding trials, (3) adjustment of pressure, time and temperature during the molding of a relevant artifact. Approach (1) is the most common way that has been used. Fluidity, pseudoplasticity, flow activation energy and yield stress are the rheological parameters that can be determined or generated from rheometer measurements. Some criteria have been established between the parameters and molding behavior. That is, the flow property of feedstocks to be injection molded is suggested to be pseudoplastic and the viscosity less than 1000 Pa.s at shear rate ranging from 100 to 1000 s^{-1}.[4] Small flow activation energy or yield stress is favorable to obtain molded parts free from defects.[6,7] However, a complete correlation between viscosity measurements and moldability is still under investigation. Approach (3) is the most realistic way. However, it requires considerable testing time and a large amount of material. On the contrary, approach (2) is more practical than approach (1) in simulating the material flow into a mold cavity. It is also more economic than approach (3) during trials. Therefore, the present work uses approach (2), i.e. a spiral flow analysis for examining the flow properties of feedstocks.

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The binder is usually a muli-component mixture for fulfilling the requirements for PIM.[8,9] Usually, the binder is composed of resin, wax and processing aids. Resin such as polyethylene or polypropylene is the major binder offering the general rheological property. Waxes as the minor binder lower the viscosity of feedstocks. Stearic acid is a lubricant commonly used in promoting the flow of material especially at high powder loading.[10,11] Coupling agents are sometimes included in binder formulations to modify some properties.[12,13] Until recently, many works have obtained useful results concerning the influence of binder composition on the rheological properties of the resulting mixtures.[9–13] However, due to the complexity of binder formulations, the effect of each component on the flow of feedstocks has not been fully studied.

The present study focuses on the above aspect. An experimental design method was applied to systematically investigate the influence of each formulation ingredient on the flow properties of alumina feedstocks through a spiral flow analysis. This method is based on a McLean–Anderson design that is good for mixture problems where ingredient concentration is equal to unity.[14]

EXPERIMENTAL

Experimental materials included alumina powder (AL), polypropylene resin (PP), paraffin wax (PW) and stearic acid (SA). The powder is of 99·8% purity (AES-11, Sumitomo Chemical, Japan). It has an average particle size of 0·4 μm, with irregular shape. Its appearance is shown in Fig. 1. Fourteen different formulations based on the McLean–Anderson method were designed and the compositions are listed in Table 1. The ranges of AL, PP, PW and SA were 87–89, 5–9·25, 3–7·25 and 0·75–1·5 wt%, respectively. The range of ingredient concentration was selected in such a way that all formulations could be injected into the molded cavity successfully. During mixing, PP was first put in a two-roll mill at 160°C until it melted. Proper amounts of AL, PW and SA were then added gradually. The mixture was mixed at the same temperature for 30 min. The material after mixing was cooled down to room temperature and was pulverized into 1–5 mm particles that served as the feedstocks. The feedstocks were then injected into a mold with a spiral flow shape through an injection machine. The injecting pressure was about 7 MPa. The barrel and nozzle temperatures were kept between 160–200°C. The spiral flow distances (SFDs) of molded samples were measured and the effect of each formulation component on SFD was analyzed.

Fig. 1. The SEM photograph of alumina powder.

Table 1. Mix proportion of each formulation

Formulation	Al_2O_3	PP	PW	SA
#1	88·000	7·875	3·000	1·125
#2	87·667	5·000	6·083	1·250
#3	89·000	7·250	3·000	0·750
#4	87·000	9·250	3·000	0·750
#5	87·000	5·000	6·500	1·500
#6	88·000	6·250	4·250	1·500
#7	87·000	6·937	4·938	1·125
#8	87·667	7·167	4·416	0·750
#9	87·000	8·500	3·000	1·500
#10	89·000	6·500	3·000	1·500
#11	87·000	5·000	7·250	0·750
#12	87·857	6·643	4·321	1·179
#13	89·000	6·250	3·500	1·250
#14	89·000	5·000	4·500	1·500

RESULTS AND DISCUSSION

Measured SFD of each formulation at different temperatures is given in Table 2. Apparently, lower SFDs were obtained for formulations (e.g.

Table 2. SFD of each formulation at three different temperatures

Formulation	SFD (cm)		
	160°C	180°C	200°C
#1	11·27	15·30	16·10
#2	18·20	20·80	32·60
#3	3·10	3·95	5·14
#4	8·32	10·75	14·80
#5	19·42	22·80	41·08
#6	14·30	18·70	25·40
#7	12·70	15·60	21·90
#8	9·90	14·20	17·50
#9	8·50	14·50	19·30
#10	3·15	4·05	6·53
#11	18·30	22·30	39·50
#12	8·17	10·60	14·65
#13	3·15	5·40	12·45
#14	3·30	7·53	13·50

#3, #10, #13, #14) with higher powder loading, as an indication of lower fluidity.[15] Furthermore, SFD increases with temperature. Detailed analyses about the effect of each ingredient on SFD will be discussed in a later section.

Figure 2 compares SFD of PP with that of #5 formulation. It is noted that PP exhibits longer SFD than this formulation although PP is more viscous than the feedstock. This is because the thermal conductivity of PP is much smaller than that of PIM feedstocks. The plastic will freeze much slower than the alumina-containing mixture when being injected into the mold.[3] As there is only 2% difference in powder concentration among our studied formulations, the thermal conductivity factor on SFD was not considered to be important.

Figure 3 shows the appearance of molded parts with spiral flow shape at three different injection temperatures. It was found that the injected part at 200°C appeared to exhibit rougher surface and more porosity than the other two. It probably results from the evaporation of some binder components of low-boiling point during injection molding. In other words, the formulations should be injection molded at lower temperature to avoid such defects.

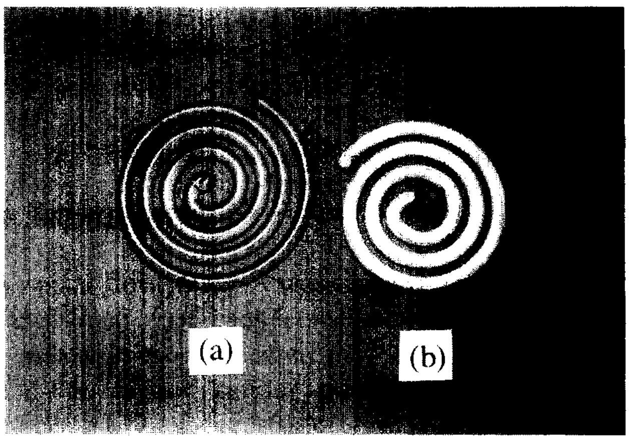

Fig. 2. The appearance of molded parts with spiral flow shape: (a) PP and (b) #5 formulation.

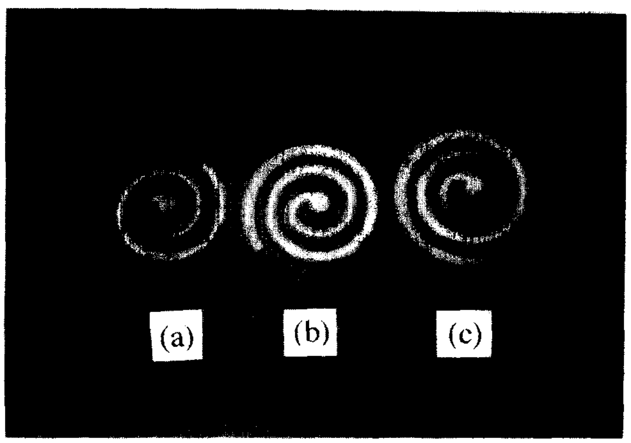

Fig. 3. The appearance of molded parts with spiral flow shape at (a) 160°C, (b) 180°C and (c) 200°C.

Linear mixture model analysis

A linear mixture model is appropriate for examining the trend and the influence of each ingredient on SFD of feedstocks. This model can be expressed as:

$$Y = B_1X_1 + B_2X_2 + B_3X_3 + B_4X_4$$

Y is the measured SFD at 160°C, X_i and B_i (i = 1–4) correspond to each ingredient and the related coefficient. Each ingredient in this model is assumed to be independent and has no interactions with others.

Figures 4–7 show the effects of each component on the SFD of feedstocks with the other ingredients kept constant. SFD was observed from Fig. 4 to decrease with AL, since the viscosity of feedstocks increases with powder loading.[3] In contrast, increase of either PP, PW or SA would increase SFD, as is illustrated in Figs 5, 6 and 7, respectively. Therefore, the influence of binders on SFD is opposite to that of the powder.

The relative effect of each binder component on SFD could also be estimated from comparing the slope of each curve in Figs 5–7. It is found that the

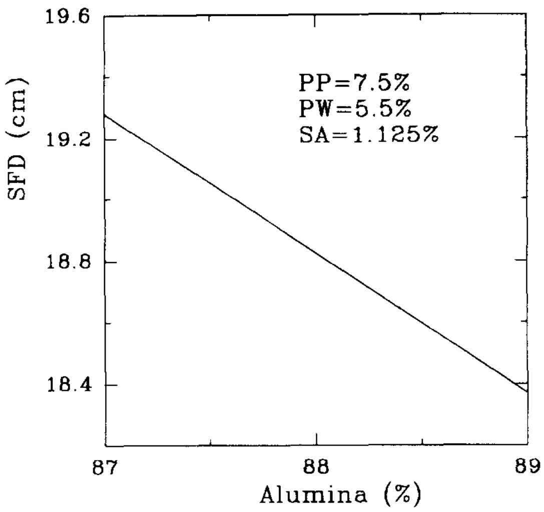

Fig. 4. The effect of AL on SFD of feedstocks.

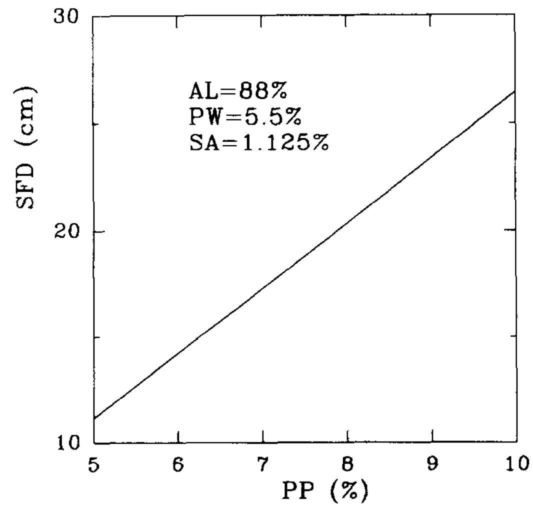

Fig. 5. The effect of PP on SFD of feedstocks.

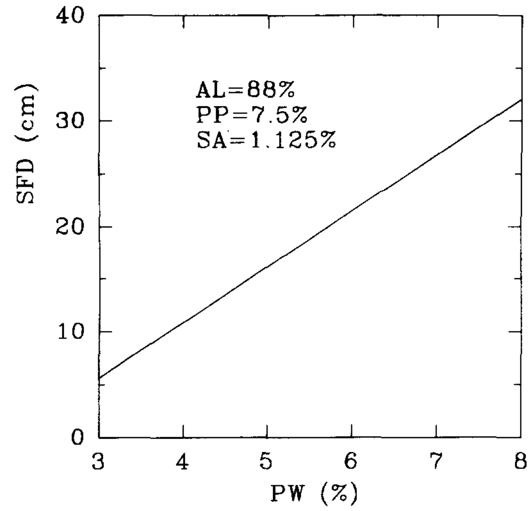

Fig. 6. The effect of PW on SFD of feedstocks.

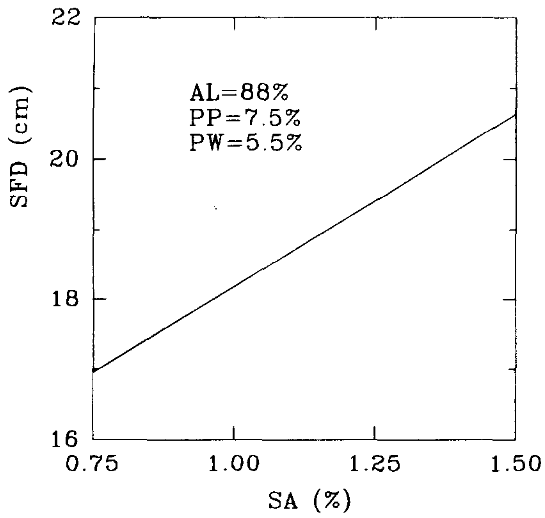

Fig. 7. The effect of SA on SFD of feedstocks.

slope of the curve in Fig. 6 is the highest, followed by those in Figs 7 and 5. Therefore, PW is the most effective component to promote the fluidity of feedstocks, SA is the next and PP is the last.

Quadratic mixture model analysis

The influence of each component on SFD was clearly demonstrated from the above results. However, it is noted that these results were generated from the analysis of a linear mixture model that neglected the interactions of each component. In order to explore the combining effects, a quadratic mixture model is further used for SFD analyses. This model can be expressed as:

$$Y = B_1X_1 + B_2X_2 + B_3X_3 + B_4X_4 + B_{12}X_1X_2 + B_{13}X_1X_3 + B_{14}X_1X_4 + B_{23}X_2X_3 + B_{24}X_2X_4 + B_{34}X_3X_4$$

where Y is the measured SFD at 160°C, X_i and B_i (i = 1–4) correspond to each ingredient and the related coefficient.

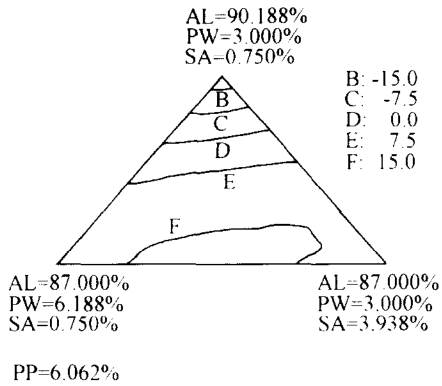

Fig. 8. The SFD contour map as a function of AL, PW and SA.

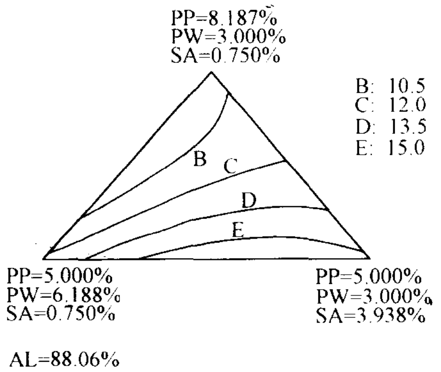

Fig. 9. The SFD contour map as a function of PP, PW and SA.

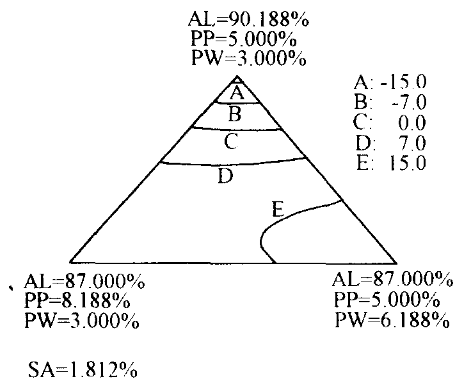

Fig. 10. The SFD contour map as a function of AL, PP and PW.

Figures 8–10 show the contour maps that illustrate how SFD behaves as a function of three ingredients at a time. In Fig. 8, PP is held at a concentration of 6·062%. All other ingredients total 93·938%. The apex of the triangle represents a mixture with 90·188% AL, 3% PW and 0·75% SA. The base represents a series of mixtures in which AL is at its minimum value of 87%, and PW and SA comprise the remaining 6·938%. On the base, PW decreases from 6·188% at the left vertex to 3% at the right, while SA increases from 0·75% to 3·938%. SFD generally increases with increasing PW or SA, or decreasing AL. At 87% powder concentration, SFD increases with PW, reaches the maximum value, and decreases thereafter. The PW/SA ratio falls between 1·37 and 4·14 while obtaining high flow distance (SFD > 15 cm). The range becomes smaller as alumina content is increased. When the alumina content reaches 88%, the PW/SA ratio is limited to a very small range around 2·16 to get such flow distance. Contrarily, for AL > 88%, SFD is increased with increasing SA or decreasing PW. A similar trend is observed in Fig. 9 as well. It means that SA is more effective than PW in enhancing the flow of feedstocks when alumina content is more than 88%. The result confirms that SA is normally utilized in formulations to reduce the viscosity of materials, especially at very high powder loading.[10] In addition, the contour lines (B, C, D) become zero or negative if AL reaches 89% or higher. That means feedstocks could not be injected into the mold cavity at such high powder loading. Interestingly, 89% by weight is about 65% by volume, which is the critical powder volume concentration obtained by Markhoff *et al.* for alumina.[16]

Figure 9 shows the effect of PP, PW and SA on SFD of feedstocks, while alumina content is fixed at 88·06%. SFD decreases with increasing PP, or decreasing PW or SA. Figure 10 shows the effect of alumina, PP and PW on SFD of feedstocks, while SA content is fixed at 1·812%. SFD increases as PP or PW is increased, or the alumina content is decreased. It is clear that both SA and PW are more effective than PP in promoting SFD.

CONCLUSIONS

A spiral flow analysis was conducted for studying the influence of each component on the flow of feedstocks. It is indicated that lower alumina content or higher binder content would provide longer SFD of feedstocks. For alumina content less than 88%, PW is most effective in enhancing SFD, followed by SA and PP. For AL > 88%, the order to increase SFD is SA > PW > PP. These observations could be helpful for adjusting the binder compositions to get proper rheological properties for successful injection molding.

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