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Influence of Powder Parameters on the Properties of Highly Filled Metal Powder Systems

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ABSTRACT

The quality & properties of the final sintered product in Metal Injection Molding (MIM) is largely influenced by the type of the powder & its properties such as particle shape, particle size distribution (PSD) packing density and the powder-binder composition. The powder considered in the study is an iron powder of particle size $d50 - 3 \mu$ (Finer grade used in MIM industry) & $d50 - 45 \mu$ (Coarser grade used in Powder metallurgy especially in press & sinter applications). Here is an attempt to compare the properties of feedstock prepared with finer, coarser, and a chosen combination of both to understand the advantages and dis-advantages of each powder including its cost benefits. The chosen powder combination was mixed with a proprietary thermoplastic binder system which acts as a matrix to hold the powder particles together. The highly loaded metal powder system varying in powder particle size and shape with same binder matrix was mixed at predetermined ratio and injection molded to tensile specimen to characterize metallurgical and mechanical properties. The molded specimens were sintered in an inert atmosphere in a batch furnace. The experimental results indicate that the finer particle size is beneficial in terms of the stability of the feedstock during injection molding, improving the sintered density, deformation behavior and mechanical properties of the compact after sintering.

Keywords: Particle shape, Particle size, Feedstock

1. INTRODUCTION

Metal Injection Molding (MIM) is relatively a new technology to manufacture small, precision and complex metal components. Smaller component size with medium to high volume production enable to manufacture highly complex products along with lower unit cost is a salient feature of this technology in comparison to other traditional manufacturing technologies like precision machining and Powder Metallurgy. Metal powders used in MIM industry ranges from 3-20 µm in mean particle size which is essential to improve the density, mechanical properties of the final sintered product after sintering [1]. Hence, the use of finer grade powders carries a premium in many of the application where it is needed to meet the mechanical properties for the higher end applications. There are applications where in minimum density and mechanical properties are of interest and therefore the addition of coarser powders provides cost benefits due to its higher yield in the powder production technique.

Heany et al.[2] has studied the mechanical properties of pre-alloyed and master alloyed gas atomized powders of particle size d50 -16 µ and d50 -22 µ respectively. Tensile specimens were molded to study the green strength, feedstock stability, sintered mechanical properties and microstructure. It was observed that sintered strength of the material produced using a prealloyed powder was higher than the master alloy powder. Little difference in mechanical properties existed between the materials fabricated using these two powders. Berenika Hausnerova et al. [3] has presented a paper on the visco-elastic properties of highly filled compounds consisting of hard metal carbides and paraffin wax, ethylene acrylic acid copolymer based binder system. Four different kinds of hard metal carbide powders differing in particle size distribution were considered to structural changes investigate during shear deformation. It was concluded that, the compounds based on powders having high portion of small particles tend to have higher visco-elastic functions, while behavior of material containing large particles remains the course of visco-elastic melt. Kipphut and German [4] indicated that flow by viscous creep during thermal debinding is a major cause of compact deformation. The ability to retain compact shape and attain uniform shrinkage depends on the inherent inter-particle friction of the powders as well as other factors. LIU et al [5] has revealed the effect of particle size and shape, solid loading on the deformation effects. Results indicate small particle size, irregular particle morphology and high solid loading are favorable to prevent deformation effects. A systematic study by Animesh Bose, Isamu Otsuka [6] on the MIM processing of ultra fine 316L powders discusses on the improvement in properties of an ultra-fine 316L powder of mean particle size 5 μ in comparison to a standard SS 316L grade of 10 µ. The investigation concludes with Ultra fine powders exhibiting lower surface roughness, higher sintering rate along with better tensile strength than conventional powders. The ultra fine powders also allowed filling of the fine details in the mold. Mukunda B N et al [7] has investigated that the low volume fraction of lubricating liquid in a feedstock favors powderbinder separation during injection molding. The study demonstrates the optimum loading for Carbonyl iron powder and 17-4PH Stainless Steel (SS) alloving composition of solid loading 59 vol% and 68 vol % respectively based on the results of stability test. Any discrepancy in apparent viscosity in constant shear rate test indicates the powderbinder separation which is observed especially at higher solid loading. The density of the

MIM 4140 is one of the most commonly used low alloy steel in MIM industry. As per the MPIF standard-35, typical yield strength and UTS of the material is 300 MPa and 750 MPa respectively. The % elongation of the material is 8 with a minimum density of 7.5 g/cc. These properties can be achieved through elemental formulation with the addition of 1% Chromium and 1% Nickel to the base Iron (Fe). The desired mechanical properties of MIM 4140 can only be achieved if there is enough surface contact between the particles which exhibits kinetics of sintering with desired sintered density.

The present aim of this research is to study and compare the rheological properties, sintered density, deformation behavior, metallurgical & mechanical properties of MIM 4140 material using a finer (Carbonyl iron Process, CIP) and coarser grade Iron (Powder Metallurgy, PM) powder having a particle size of d50 -3 μ and d50 -45 μ respectively at a chosen powder loading. The study was also extended to understand the properties of

MIM 4140 material with 25 wt % of coarser and 75 wt % of finer grade powder and cost comparison was performed among the chosen powder formulations.

2. EXPERIMENTAL PROCEDURE

Iron powder manufactured through carbonyl iron process and water atomization technique was the major powders used in the present study. The chosen powder formulation was mixed with a thermoplastic wax based binder and feedstock was prepared. Scanning electron microscopy (SEM) showed a spherical shape for CIP and irregular nodular morphology for PM grade powder as shown in Figure 1 and Figure 2 respectively. Powder characteristics of the chosen powder combination are as reported in Table 1. A standard grade of MIM 4140 was formulated to study and compare the rheology, deformation behavior, metallurgical and mechanical properties of the feedstock.

PowderParticle size d50, (μm)Particle shapeTap density (g/cc)CIP grade3Spherical4.28PM grade45Irregular3.94		ental propertie		
CIP grade3Spherical4.28PM grade45Irregular3.94	Powder	Particle size d50, (µm)	Particle shape	Tap density (g/cc)
PM grade 45 Irregular 3.94	CIP grade	3	Spherical	4.28
	PM grade	45	Irregular	3.94



Figure 1: SEM micrograph of CIP grade powder.



Figure 2: SEM micrograph of PM grade powder.

Torque rheometer was used to evaluate the Critical Powder Loading by measuring the torque of the feedstock with reference to time at constant temperature. All the chosen feedstock combination in the present study was tested at 180°C. The feedstock was loaded into the chamber while the rotors are running. After achieving the initial torque for every powder loading, the next dosage of powder (in vol.%) were loaded. Any erratic fluctuations in torque value indicate the critical powder loading.

The feedstock was prepared using double blade sigma mixer containing rectangular blades at $180 \, {}^{0}C$. The blades pass through every point in the vessel at 20 RPM promoting homogeneous mixing between a powder and binder. The chosen feedstocks were granulated after mixing.

Capillary Rheometer was used to evaluate powderbinder separation by measuring apparent viscosity of the feedstocks with reference to different time intervals at constant temperature and shear rate (300 1/s). The Capillary rheometer consist of a barrel, temperature controlled heater surrounding the barrel and a piston pushing the material through the barrel at different speed. The feedstock was loaded in to the barrel of the capillary Rheometer and apparent viscosity was measured at every 60 seconds for the interval of 15 minute to understand the change in apparent viscosity. All the chosen feedstock combination in the present study was tested at 180 °C. Feedstock viscosity should be uniform with very little or no scatter if the material doesn't separate over a specified time intervals. Any erratic fluctuation in apparent viscosity over time indicates unstable behavior of the feedstock. Pycnometer density was measured for virgin feedstock, feedstock left over in the barrel after the test (plug) and extrudate. If higher the variation in density between the virgin and plug or extrudate, then such feedstocks are interpreted to cause powder-binder separation during molding.

The chosen feedstock combination was molded in injection molding machine with a standard tensile bar (MPIF 35) and a rectangular specimen of dimension 40x10x1.6 mm. The molded parts were thermally debound and sintered in a vacuum furnace under nitrogen atmosphere. Shape retention analysis was done using a rectangular specimen of dimension 40x10x1.6 mm as shown in the Figure 5. The specimen with highest deformation tends to deform higher in sintering and vice versa.

3. RESULTS & DISCUSSION

The experimental powder loading was decided based on the Critical solid Loading (CSL) using Torque rheometer. It was found that optimum loading for any powder-binder mixture will be 5-7% lower than the CSL for any highly filled feedstock [8]. In the present study, CSL of 67 vol.



Figure 5: Sintered and Green parts of Tensile and Rectangular specimens

Table 2.	Results of	Critical	Solid	Loading	(CSL)
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Feedstock	Critical Solid Loading, (CSL), Exp. (Vol. %)
CIP	67
PM	58
CIP+PM	65

% was arrived using torque rheometer for CIP feedstock. Hence, 59 vol% of powder loading was chosen to be an optimum powder loading for CIP feedstock. The results of CSL of the chosen powder combinations are presented in Table 2. CSL of 58 vol. % was observed for feedstock prepared with PM grade powder as shown in the Table 2. The reduction in CSL value of PM feedstock is due to the irregular morphology of the PM grade powder which degrades the packing characteristics of the powder particles and reduces the critical powder loading [1].

The feedstocks were prepared with the chosen powder loading and viscosity measurement was performed using capillary rheometer as shown in Table 3. The viscosity of CIP feedstock was observed to be 109.81 Pa.S in comparison to the feedstock prepared with the PM grade powder. The increase in viscosity in CIP feedstock is due to the inter particle friction between the powder particles which increases the viscosity of the feedstock. The viscosity of CIP+PM combination found to be higher viscous than the feedstock produced with the pure PM grade powder. The chosen feedstock formulation was also tested for stability using constant shear rate test and the results are as reported in Figure 4. The decrease in viscosity was observed with time interval of 15 minutes with CIP and CIP+PM powder based feedstock whereas increase in viscosity was observed in PM based feedstock. Density of virgin, plug density (feedstock left in the barrel of the capillary rheometer after the constant shear rate test) and extrudate density was measured. The percentage

Feed-	Exp.	Viscosity	Stability	Distortion
stock	powder	in Pa.S		(mm)
	loading			
	chosen			
	(vol. %)			
CIP	59	109.81	Stable	1.14
PM	50	38.50	Un-	5.12
			Stable	
CIP+PM	1 59	72.15	Stable	2.87

Table 3: Feedstock characteristics

variation in density of plug and extrudate with reference to the virgin feedstock was calculated and results are reported as shown in Figure 9. The % variation in density of PM based feedstock found to be highest of 3.5 % which indicate there is an occurrence of powder-binder separation in PM based feedstock. The density variation found to be in-significant in case of CIP and the CIP+PM



Figure 4: Stability of feedstock formulation chosen.



Figure 9: % Variation in density wrt Virgin Vs Feedstock.

mixture with very minimal or no powder-binder separation. From Figure 4 and Figure 9, it can be clearly seen that CIP+PM feedstock found to be most stable in terms of viscosity with least density variation among the chosen formulation. It is due to the addition of 25 wt. % of irregular PM grade powder particles which improves the stability of the feedstock with better particle interlocking which is in agreement with the previous studies [5]. The feedstocks were injection molded into tensile specimens and rectangular test specimen of dimension 40x10x1.6 mm as shown in Figure 5.

The deformation behavior of the chosen feedstock formulation was tested as shown in Figure 3. Results reported in Table 3 indicate CIP feedstock has got the least distortion in comparison to the PM feedstock and the CIP+PM combination chosen in the study. This is due to the finer powder particle size of the CIP particles which increases the particle contact reducing the interparticle spacing which reduce the distortion in the components [7]. PM feedstock showed highest distortion of 5.12 mm due to the higher particle size of PM grade powder which enhances the interparticle spacing which in turn results in higher distortion. With the addition of 75 wt % CIP grade finer particles to the 25 wt. % of courser PM powder particles, the distortion has improved from 5.12 mm to 2.87 mm due to better interparticle contact between the particles.



Figure 3: Deformed Rectangular specimens after sintering.

Mechanical properties such as yield strength, ultimate tensile strength, hardness and percentage elongation for all the chosen feedstock formulation was tested using tensile specimen and the results are as shown the Table 4. The yield strength and UTS of CIP feedstock was found to be the highest in CIP feedstock whereas feedstock made of PM grade powder found to be the lowest strength among the chosen formulation. The mechanical properties of 25 wt. % addition of PM grade courser powder found to be lower in strength in comparison to the CIP feedstock. The chosen feedstock formulation was also tested for percentage porosity and density of the sintered specimens and results are as shown in the Table 5. Feedstock made of CIP powder found to be lower in porosity of 2.03 % with density of 7.61 g/cc in comparison to the PM based feedstock with porosity of 38.5% and density of 7.13 g/cc. This is because of the lack of proper bonding between the particles in PM grade powder even after sintering due to which higher porosity is observed with lower density & mechanical properties. The theory was also supported with microstructure analysis as shown in Figure 7. The microstructure analysis of the chosen CIP, PM and CIP+PM combination is as shown in Figure 6, 7 and 8 respectively. It was also observed that density may not increase in PM grade powder over some point of time due to higher particle size and hence addition of alloying elements such as Copper may help to improve properties up to certain extent. Feedstock with CIP+PM combination found to have vield strength and UTS meeting MPIF standards as shown in Table 4 whereas sintered density found to be 7.43 g/cc which is quite less than the required minimum density of 7.50 g/cc.

Table 4: Mechanical properties of MIM 4140

Feed- stock	Yield strength (MPa)	Ultimate tensile strength (MPa)	% Elongation	Hardness
CIP	726.85	920.98	6.49	21.24
PM	103.95	116.56	0.95	20.5
				HR15N
CIP+PM	509.12	750.52	7.05	21.08
				HRC

Table 5: Sintered Density and Porosity of MIM4140

Feed- stock	Exp.pow der loading (vol. %)	Sintered density (g/cc)	% Porosity
CIP	59	7.61	2.03
PM	50	6.66	38.5
$\operatorname{CIP} + \operatorname{PM}$	59	7.43	3.52

Figure 10 shows the yield strength and cost comparison of all the three chosen combination. As shown in the figure, CIP feedstock found to be highest in terms of yield strength and cost of the powder in comparison to the other two combinations. Feedstock based on PM found to be the lowest in terms of cost ('X') with mechanical properties far below the expectation as per the standard and hence not suitable for MIM application. While addition of 25 wt. % of PM grade powder to the 75 wt. % of CIP powders reduced the cost of the feedstock up to 22% in comparison to the standard CIP grade powder with slight reduction in mechanical properties as shown in Table 4. Hence, it can be concluded that the derived CIP+PM combination can be used in

application where it doesn't justify the requirement of higher mechanical properties but cost is the limiting factor.



Figure 10: Yield strength and cost comparison.



Figure 6: Microstructure of CIP feedstock.



Figure 7: Microstructure of PM feedstock.



Figure 8: Microstructure of CIP+PM grade.

4. CONCLUSION

The present study demonstrates the importance of using finer powders in manufacturing complex, precision, small metal components in MIM industry today. MIM 4140 with finer CIP powder found to be the best in terms of mechanical properties with least distortion in sintering in comparison to the courser powder used in PM industry today. The courser powders tend to exhibit unstable behavior in constant shear rate test along with higher deformation and lower mechanical properties which found to be not suitable for MIM application. However, addition of 25 wt. % of courser irregular PM grade powder to the 75 wt. % of CIP powder improves the stability of the feedstock when compared to the pure CIP based feedstock. It was also found that there is an enormous improvement in the deformation resistance of the compact along with mechanical properties in the CIP+PM mixture in comparison to the pure PM based feedstock. The addition of 25 wt. % of PM grade powder to the CIP powder also reduced the cost of the feedstock up to 22% with little depreciation in mechanical properties. Hence, the derived CIP+PM combination can be used in application where cost is the main criteria for the end user.

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