Innovative process to die compact injection molding powders

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ABSTRACT

A new economical and innovative process has been developed to die compact injection molding (spherical) powders with low amount of polymer (1 wt. %). The successful compaction of spherical particles was achieved due to uniform coating of the polymer on the particles. The process uses polymer in emulsion form in water. A brief description of the process is presented in this paper. Initial compaction studies carried out with admixed powders of 316L stainless steel and 1 wt. % polymer showed maximum green densities up to 84 % theoretical at compaction pressure of 1380 MPa (200 kpsi). The compacted samples were successfully sintered to near full density (>96% theoretical density) after heating to 1300˚C for 60 minutes in a hydrogen atmosphere.

INTRODUCTION

Powder metallurgy technology deals with manufacturing of components from metal/ceramic powders. The process consists of three primary steps i.e. shaping, polymer removal and sintering. In the shaping stage, the powders are formed to the required component shape either by use of external pressure (die compaction) or by utilizing the flowability property of the powder-polymer mix (injection molding).

Conventionally irregular shaped powders are used for die compaction. The irregular shaped powders undergo mechanical interlocking during compaction process thus providing strength to the compacted sample. Usually less than 2 wt. % polymer is used in this process. The polymer is used for providing lubrication. In case of injection molding process small spherical shaped powders are used as the initial raw materials. The spherical powders are mixed with large amount (more than 15 wt. %) of polymer. The metal – polymer mix (feed stock) is used for shaping the component.

Various techniques like high velocity compaction [1-3], warm compaction [4-6] and double press-double sinter [7-9] have been developed to make components with high sintered densities and superior properties. The driving force for sintering process is the reduction in surface energy of the particles [10]. Irregular shaped powders due to their low surface energy requires high sintering temperatures. The use of small spherical particles for die compaction will lower the sintering temperatures and aid attaining high sintered densities at low temperatures. However die compaction of spherical powders is difficult due to the inherent shape of the powders. The spherical shape of the powders offers no interlocking of the particles during die compaction thus offering no strength to the die compacted sample. In this paper an innovative process developed to die compact spherical powders with low amount of polymer is presented.

EXPERIMENTAL

Gas atomized 316L stainless steel powders of average particle size of 9.3 μm were used to verify the process developed in the study. The characteristics of the as-received powder are summarized in Table 1. The scanning electron micrograph (SEM) of the as-received powder is shown in Figure 1. The SEM shows the spherical shape of powders which is typical for gas atomized powders.

Powder property	Gas atomized 316L stainless steel
Particle size distribution D_{10} D_{50} D_{90}	 5.4 ± 0.3 % 9.3 ± 0.2 % 14 ± 0.1 %
Apparent Density (g/cm^3)	4.3
Tap Density (g/cm^3)	4.9
Pycnometer density (g/cm^3)	7.9

Table 1. Summary of characterization results of as-received powders.

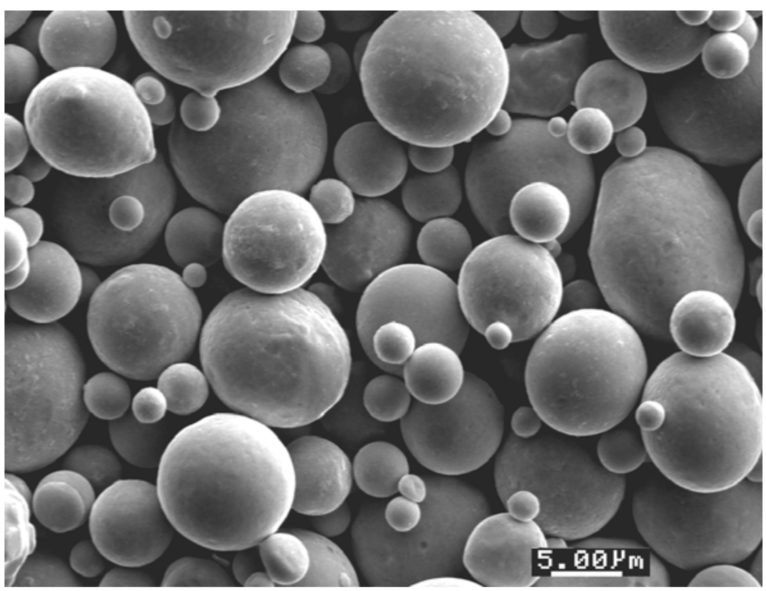

Figure 1: Scanning electron micrograph of as-received gas atomized 316L stainless steel powder.

The outline of the innovative process developed to die compact small spherical powders is shown in Figure 2. Initially, polymer emulsion corresponding to the final required amount of polymer is placed in a glass beaker. The emulsion is diluted by adding distilled water of five times its weight. The solution is stirred and metal powders are then added to the diluted emulsion. The diluted emulsion with the metal particles is further stirred. A slurry of metal particles and diluted polymer is obtained at this stage. In the next step, the slurry is dried at 100˚C in an oven to remove the water. The dried mix is fed into in a granulator to obtain admixed metal powders with polymer. The admixed powders are then sieved to break the soft agglomerates. -100 mesh powders are used for compaction. The admixed powders were then compacted to cylinders of diameter 12.7 mm and height of 4 mm. The compaction of the powders was carried out on a 60 ton hydraulic press.

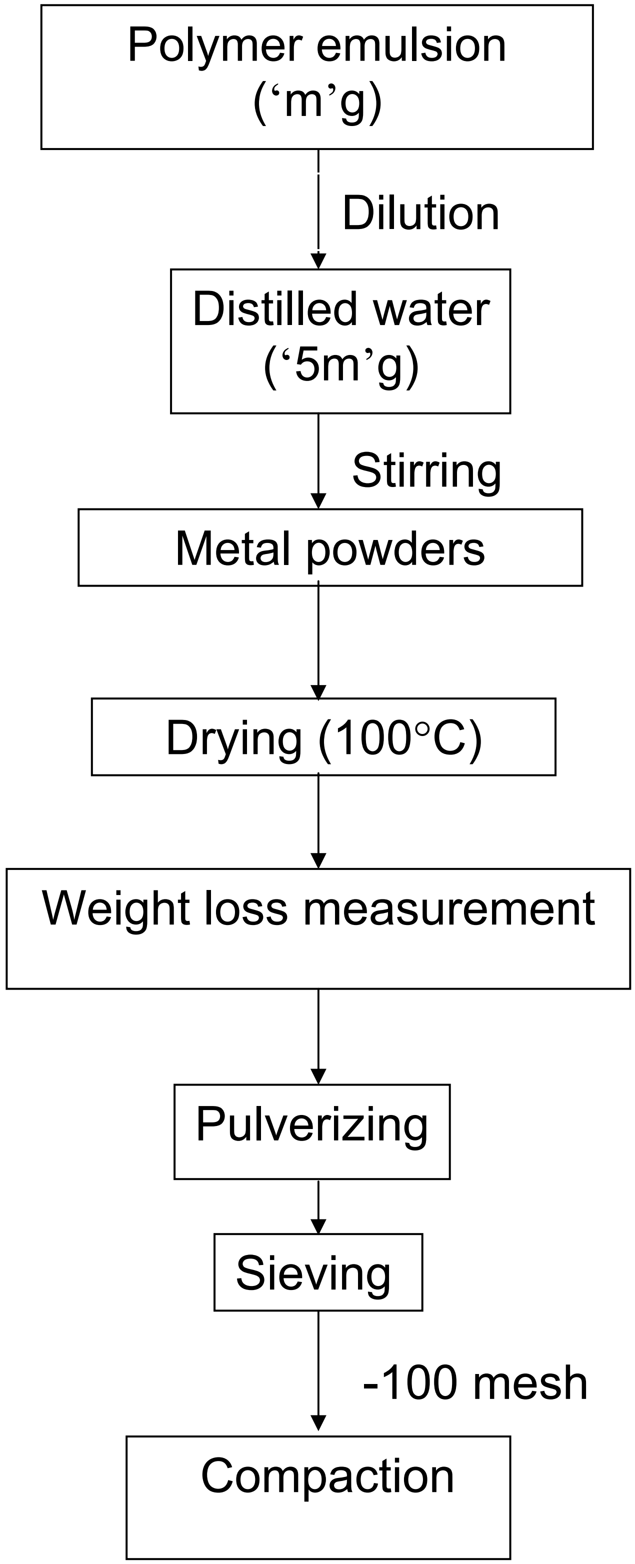

Figure 2. A brief outline of the new process developed to die compact gas atomized powders.

RESULTS AND DISCUSSION

The repeatability of the process developed was verified by measuring the carbon content of the admixed powders mixed with 1 wt. % polymer prepared from three different batches. The measured carbon content of these powders is shown is Table 2. The results show good repeatability of the process.

Number	Carbon content (wt. %)	Standard deviation
Batch -1	0.52	0.07
Batch -2	0.58	0.00
Batch -3	0.54	0.01

Table 2. Carbon content of admixed powders mixed with 1 wt. % polymer from three different batches.

The results obtained from the compaction study from admixed powders with 1 wt. % polymer is shown in Table 3. The admixed powder showed good compactability and handling strength. A maximum green density of 84 % theoretical was achieved at compaction pressure of 1380 MPa (200 kpsi). The compaction of the admixed powders were carried out without addition of any die wall lubricant. The green cylinders showed no exterior defects like cracking or delamination.

Pressure MPa (kpsi)	Green density (g/cm^3)	% theoretical
526 (76)	5.6	70
965 (140)	6.4	80
1380 (200)	6.7	84

Table 3. Green density obtained during compaction studies on admixed powders of gas atomized 316L stainless steel and 1 wt. % polymer.

Sintering of the samples was carried out in hydrogen atmosphere at various temperatures. The sintering results of the cylinders are shown in Table 4. A maximum density of 97 % theoretical was obtained on sintering the samples to 1300˚C for 60 minutes. The results show the requirement of lower temperature to sinter the powders to high densities. Conventially 316L powders are sintered with addition of boron and heating to a temperature above 1400˚C [11,12].

Heating cycle	Sintered density (g/cm^3)	% theoretical
5˚C/min to1300˚C, 1h	7.7	97
5˚C/min to1350˚C, 1h	7.8	98
5˚C/min to1400˚C, 1h	8.0	100

Table 4. Results obtained from sintering study.

The scanning electron micrograph of the fractured transverse rupture bars (31.8 mm x 12.7 mm x 5-7 mm) in the green state is shown in Figure 3. The micrograph clearly shows a uniform coating of polymer on the metal particles. The good compactability of the admixed powders was attributed to this uniform coating of the polymer obtained form the developed process. The principle behind the successful compaction of gas atomized powders from the process developed is shown in Figure 4. Water atomized powders which are irregular shape undergo interlocking during die compaction and provide handling strength to the shaped component. Gas atomized powders due to their inherent spherical shape forms no interlocking during die compaction thus providing no strength to the compacted sample. The formation of a coating of polymer in the current process resulted in providing handling strength to the die compacted sample.

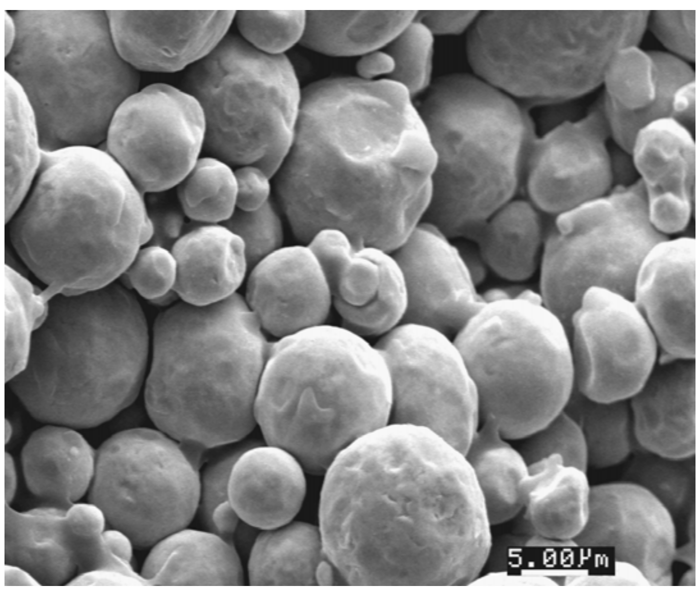

Figure 3. SEM image of fractured sample in green state showing a uniform coating of polymer on the spherical metal particles.

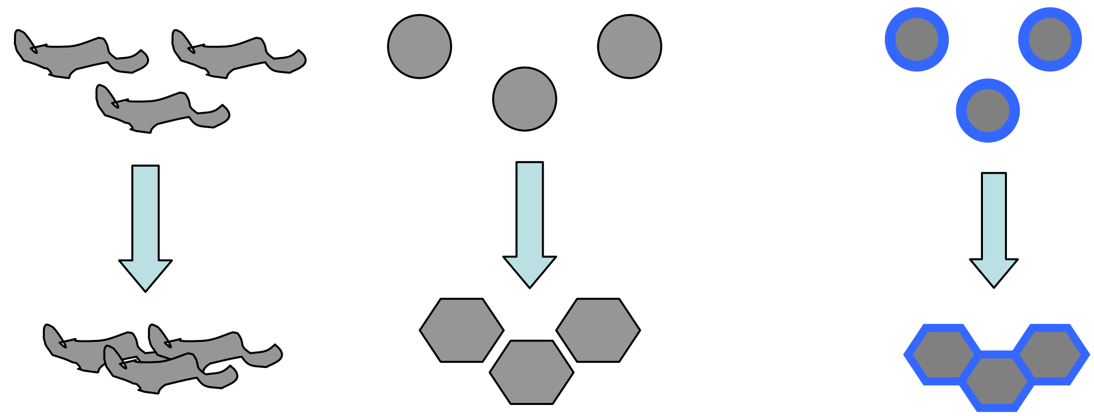

Figure 4. Schematic of the principle behind the successful compaction of admix gas atomized powders prepared from the process developed in the current study.

The advantages of the new process developed are as follows:

Good compactability and handling density with low amount of polymer:
The admixed spherical powders showed good compressibility and green strength. A maximum green density of 84 % was attained during initial experiments carried out on 316L spherical powders and 1 wt.% polymer at compaction pressures of 1380 MPa (200 kpsi)). The amount of polymer used in this process is within the limits of actual amount of polymers currently used in die compaction process.

Uniform distribution of polymer:
The polymer used in this process is supplied in the form of an emulsion in water. The process developed results in uniform distribution of polymer in the form of a coating on the metal particles.

No die wall lubrication:
The compacts were die compacted without use of external die wall lubrication. The polymer used in this process fulfilled both the functions of a binder (holding the spherical particles together) and of lubricant (reducing the friction between powders and die walls)

No defects in die compacted samples:
The components were compacted without any macro defects in the form of cracking or delamination and had a very good surface finish.

Minimum green density gradients:
The shaping of the component during compaction takes place primarily due to the deformation of the polymer. The scanning electron microscopy (SEM) images of the die compacted samples revealed partial deformation of spherical metal particles at high compaction pressures1380 MPa (200 kpsi). The minimal deformation of the metal powders will lead to low green density gradients in the compacted sample.

Increase in strength during polymer removal:
One of the drawbacks of using spherical powers in powder metallurgy process is the loss in compact strength during polymer removal stage. As the polymer is removed the spherical particles due to lack of interlocking as in the case of irregular powders provide no strength to the component thus resulting in distortion and shape loss. However in the case of present system, it was observed the polymer was stable until high temperature where the initial bonding of particles due to sintering occurs. It was also observed that the strength of the components actually increased during the polymer removal process. The increase in the strength is attributed to the burnout properties of the polymer. It was observed experimentally that distortion of the component that occurs due to polymer softening during initial stages of polymer removal is recovered once the polymer starts degrading. The polymer provides support to spherical particles till the bonding of the particles occurs due to sintering thus minimizing distortion during polymer burnout process.

Low sintering temperatures:
As mentioned above, the use of spherical metal powders will increase the driving force for sintering thus allowing the sintering of the component to take place at low temperatures. The reduction in sintering temperature will help in faster production cycles and minimizing sintering costs.

Minimum distortion during sintering:
Due the low green density gradients, the distortion of the component will be minimized during sintering process.

Economical and easy for production:
The new process developed is very easy to adapt in the manufacturing environment with minimum expenditure. The polymer used in this process is inexpensive as compared to the currently used lubricant for the die compaction process.

Application for nano powders:
The process developed can find applicability for die compacting nano powders. Nano powders are spherical in shape and are very a hard to compact due to their inherent shape and high strength. The present process can help in die compaction of nano powders thus helping in fabricating components from nano powders.

CONCLUSIONS

A innovative process was developed to die compact spherical powders with low amount of (<1 wt. %) polymer. The process was developed based on a water based emulsion polymer. The admixed powders showed good compactability and the compacted samples possessed good handling strength. A maximum green density of 84 % theoretical was achieved on compacting the admixed powders with 1 wt. % polymer at 1380 MPa (200 kpsi). The green samples were sintered to near theoretical density on heating to 1300˚C for 1h in hydrogen atmosphere. The process developed helps in reducing the sintering costs due to decrease in the sintering temperatures.

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