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Sintering schedule for warm formed iron powder compacts

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ABSTRACT

This paper reports the effects of sintering schedule to the mechanical properties and microstructure of warm formed powder compacts. Iron powder ASC 100.29 was used as main powder constituent whereas zinc stearate was used as lubricant. The premixed powder mass was compacted at 180ºC by applying 130 kN axial loading and sintered in an inert gas fired sintering furnace at different sintering schedule. The sintered samples were characterized to evaluate their mechanical properties and microstructures. The effect of sintering schedule was studied in terms of mechanical properties, focusing in particular on the relative density, flexural strength and hardness. The microstructure analysis was performed to determine the pore shape, size and distribution. The results revealed that the mechanical properties and microstructures of sintered products were affected by the sintering temperature, holding time as well as the heating/cooling rate.

Introduction

Warm compaction process has shown to provide increased density in ferrous powder metallurgy parts. This improvement in density contributes significantly to mechanical properties and thus the overall performance of the part. The combination of increased density with high performance material selections provides parts that can exceed the performance of forged or cast material counterparts while taking advantage of powder metallurgy's net shape forming capabilities [1-3].

During the forming of powder mass to a predesigned shape, a significant friction force is generated which is the major cause of density variation and also affects the component deformation. Inter-particle and particle to die wall friction also hinder pressure transmission and therefore produce density gradients inside the compact. The friction also generates significant amount of heat that affects the compaction process generally, and material properties specifically. Therefore, there is a need to use lubricant as friction reduction agent [4-6].

Sintering is a processing technique which is performed to produce density-controlled materials and components from metal or ceramic powder by applying thermal energy [7-10]. It also acts as a thermal treatment process in powder metallurgy. Small particles of a material are bonded together by solid-state diffusion during the sintering process. As material synthesis and processing become crucial in recent years for materials development, the importance of sintering is increasing as a part of metal processing technology through powder route. Unlike other processing technologies, various processing steps and variables are required to be considered for the production of high quality sintered parts. For example in the shaping step, one may use simple die compaction, isostatic pressing, slip casting or injection molding, according to the shape and properties required for the end products [11].

The sintering conditions and also the sintered properties may vary considerably depend on the shaping techniques used [12]. Various techniques and processing variables in sintering step can give variations in sintered microstructure and properties. In general, sintering process aims to produce sintered parts with designed microstructure through the control of sintering variables. Microstructural control means the control of grain size, sintered density, size and distribution of other phases including pores. In most cases, the final goal of microstructural control is to prepare a fully dense body with a fine grain structure [13].

Review in the existing literature in powder metallurgy indicates that the mechanical properties as well as the microstructure of the final product can be affected by the sintering schedule i.e., sintering temperature, heating and cooling rate, and holding time. Although some researchers have gone through this area, there is no published information on the proper sintering schedule for the production of high quality, near-net shape components from iron powder mixed with lubricant and formed at above ambient temperature. Therefore, the objective of this paper is to investigate the effect of sintering temperature, holding time and heating/cooling rate together with zinc stearate addition to the mechanical properties and microstructures of sintered iron-base components formed through warm compaction.

Materials and Method

A lab scale warm compaction rig was designed and fabricated (Figure 1) which enabled the generation of green compacts at the designated temperature, i.e., 180ºC. The main powder used in this project is iron powder ASC100.29 and zinc stearate ($C_{36}H_{70}O_4Zn$) powder was selected as admixed lubricant. The as-received iron powder has the particle size range of 20-180 μm. Two types of green compacts were prepared, i.e., green compacts without and with zinc stearate. In the case of green compacts with lubricant, the feedstock was prepared by mechanically mixing iron powder ASC 100.29 with 0.4 wt% of zinc stearate for 30 minutes. The whole experiment is divided into three sections which are green compact generation, sintering of green compacts in argon gas fired sintering furnace, and characterization of final products through mechanical testing and microstructure evaluation.

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Figure 1.Compaction Rig with Heating System

Green sample generation consists of four consecutive steps, i.e., die filling with premixed powder mass, heating of powder mass together with the die assembly, simultaneous compaction, and ejection of green compacts from the die cavity by means of bottom punch. The defect-free green compacts were then sintered in controlled environment, i.e., at heating/cooling rates of 5°C/minute and 10°C/minute, holding times of 30 and 60 minutes. All the samples were sintered at 850°C, 900°C, and 990°C. The relative densities of the sintered products were measured through dimensional measurement and the hardness of the compacts was tested through Rockwell hardness tester. The strengths of the products were measured through three-point bending test (ASTM E290-09) and the microstructures were evaluated through Scanning Electron Microscopy (SEM).

Results and Discussions

Figure 2 shows the relative density of samples sintered for 30 minutes. The result shows that the density increases as the sintering temperature increases. The figure also shows that the relative density of samples with lubricant is higher compared to that of without lubricant. The reason is that, at the compaction stage, after exposure and absorption of heat from outside, large number of new open spaces is generated among the powder particles when the lubricant broke down [14]. This mechanism allows the compaction pressure to push the particles into the gap left by the zinc stearate particles, thus produced higher relative density even after sintering.

Figure 3 shows the relative density of samples sintered for 60 minutes. Comparing to the previous results (Figure 2), the relative density of samples sintered for longer time is found to be higher. As mentioned in [15], particles diffusion is dependent on sintering temperature and time. At higher sintering temperatures, a denser structure is formed due to higher diffusion rates. Also, more diffusion can occur at longer holding time. Hence, higher density can be achieved at higher sintering temperature and longer holding time. Another parameter involved in this experiment is heating/cooling rate. From Figure 2 and Figure 3, it can be observed that slower heating/cooling rate gives better density to the sintered parts, either with or without the addition of zinc stearate. However, the influence of this parameter is not very substantial.

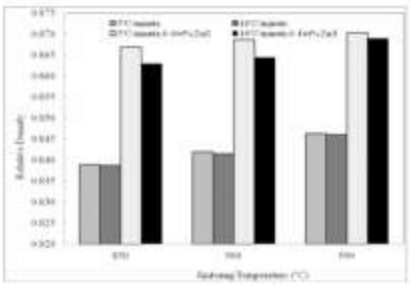

Figure 2.Relative Density of Parts Sintered for 30 Minutes

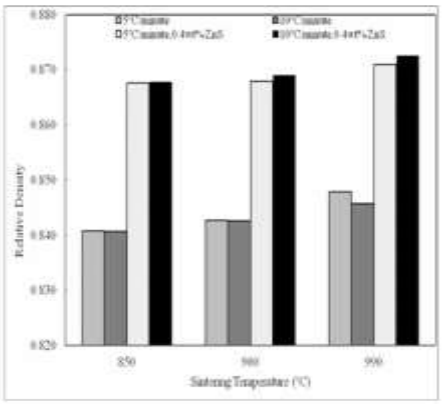

Figure 3.Relative Density of Parts Sintered for 60 Minutes

The strength variation of the samples sintered for 30 minutes is presented in Figure 4. From the figure, it can be observed that the strength of the samples, both with and without the addition of 0.4 wt% zinc stearate, increases with temperature. It is obvious that sintering at high temperature improves the strength, owning to higher relative density. The strength of the samples also improves at faster heating rate, i.e. 10ºC/minute. As reported in [16], grain growth is inhibited at faster heating rate, thus products become stronger. This concurs with the Hall-Petch theory which states that strength increases with the decrease of grain size.

On the other hand, Figure 5 shows the strength variation of samples sintered for 60 minutes. It can be observed that the strength of samples sintered for longer time is slightly lower compared to Figure 4. This is because longer holding time gives larger grain size and thus lowers the strength [15]. However, both figures show that the strength of the samples has drastically increased with the addition of 0.4 wt% zinc stearate. During the compaction process, zinc stearate functions by sliding towards the die wall from between the powder mass. This mechanism leads iron particles to move easily by reducing inter-particle friction and also minimizing the die wall friction, thus increasing the green density. Furthermore, improvement in density contributes significantly to the mechanical properties, e.g. strength, of the sintered part and thus its overall performance [17]. These explain the drastic increase in flexural strength as the powder was mixed with zinc stearate.

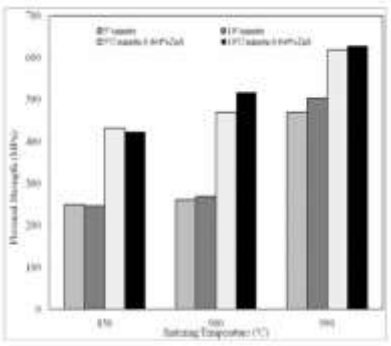

Figure 4.Flexural Strength of Parts Sintered for 30 Minutes

Figure 6 shows the SEM images of the samples sintered for different time. It can be observed at the sample sintered for 30 minutes that the contact regions among particles are still visible (Figure 6(a)). However, these contact regions are not visible in the case the sample was sintered for 60 minutes. The visible contact regions among particles are the necks between the adjacent grains created during sintering, which improved the strength of the material when subjected to bending load. The

reason is that the intergranular fracture needs additional stress to propagate by breaking the necks [18]. The absence of these necks leads to lower strength possessed by the sample sintered for 60 minutes.

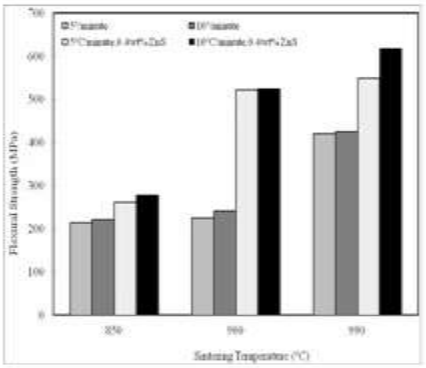

Figure 5.Flexural Strength of Parts Sintered for 60 Minutes

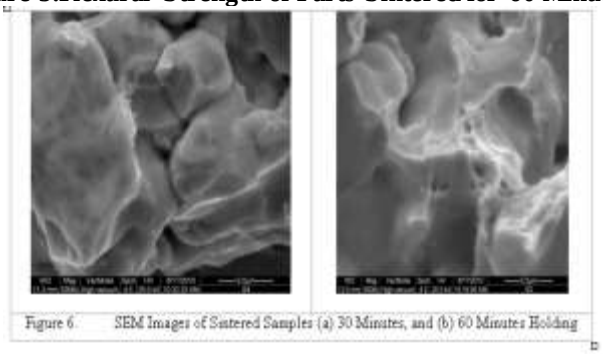

Figure 6 SEM Images of Sintered Samples (a) 30 Minutes, and (b) 60 Minutes Holding

Figure 7 shows the SEM images of samples which are sintered at 990ºC, 5ºC/minute for 60 minutes (holding time) but with and without zinc stearate. Figure 7(a) shows the microstructure of sample without zinc stearate, while Figure 7(b) shows the microstructure of sample with 0.4 wt% zinc stearate. Pore clusters and angular shape pores can be observed in Figure 7(a), while more homogeneous distributed and round pores can be observed in Figure 7(b). As mentioned in [19], the distribution of the pores is also important, since it has been shown that plastic deformation may initiate at pore clusters because of the higher localized stress intensity associated with these defects.

Vedula and Heckel [20] stated that highly localized slip bands formed at the sharp tips of angular pores producing uneven distribution of strain around angular pores. This resulted in highly localized and inhomogeneous plastic deformation compared to the deformation around round pores which was much more homogeneous. This results in higher strength recorded by samples with 0.4 wt% zinc stearate as shown in Figures 4 and 5.

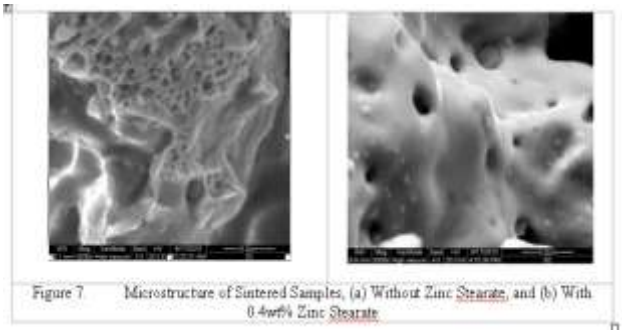

Figure 7 Microstructure of Sintered Samples, (a) Without Zinc Stearate, and (b) With 0.4wt% Zinc Stearate

Figure 8 shows the results obtained by the Rockwell hardness test performed on the samples sintered for 30 minutes. Better hardness can be observed at the samples which are sintered at 900ºC. Increasing the sintering temperature to 990ºC does not improve the hardness. It is known that high temperature promotes grain growth which can lower the hardness. This is because growth in grain size has the opportunity to occur at high temperature. It can also be observed that high heating rate improves the hardness of the samples. The reason is that the sample had shorter exposure time during higher heating rate compared to slower heating rate [21]. Thus, it gives less grain growth and better hardness can be achieved.

Result of hardness test conducted on the samples sintered for 60 minutes holding time is shown in Figure 9. The result shows similar trend as in Figure 8. From both figures, it can also be observed that the lubricated samples possessed lower hardness compared to pure iron samples. This might be due to the movement of zinc stearate particles toward the outer region of the green compact which causes lower dense surface. This surface is easily deformed when subjected to external force (low in hardness), which in this case is the Rockwell indenter.

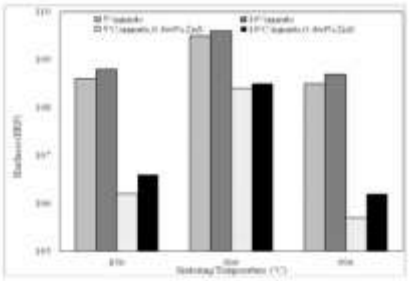

Figure 8 Hardness of Parts Sintered for 30 Minutes

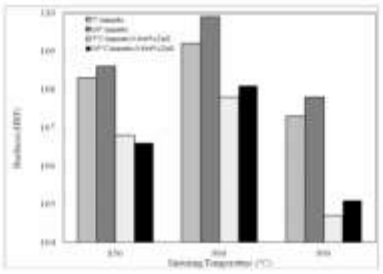

Figure 9 Hardness of Parts Sintered for 60 Minutes

The dimensions of the compacts at green state and sintered state are also been taken into concern. Dimensions of the samples are measured using digital verniar caliper which are then taken to calculate the volume. It is found that the percentage of dimensional change, i.e. shrinkage, is very minimal which almost cannot be seen by naked eyes. The percentage of shrinkage is recorded to be around 0.3 to 3.4 %. However, for samples with 0.4 wt% zinc stearate, the calculated dimensional change shows swelling. The percentage of swelling is very minimal which is around 0.2 to 1.3 %.

Conclusions

In this study, the effect of sintering parameters toward the mechanical properties of sintered parts without and with the addition of 0.4 wt% zinc stearate was investigated. High sintered relative density can be achieved by putting it in high sintering temperature, slow heating/cooling rate and longer holding time. The relative densities of the sintered parts are greatly improved by the addition of 0.4 wt% zinc stearate. The flexural strength of sintered parts can be achieved by high sintering temperature, fast heating/cooling rate and shorter holding time. Higher density gives better strength. The hardness of sintered parts is observed to be increased as the

sintering temperature increased from 850°C to 900°C, but did not improve any further as it is sintered at 990°C. Shorter holding time and fast heating rate give better hardness. Microstructure analysis shows that sintering process creates necks between adjacent particles which improve the parts' strength. Parts with homogeneous distribution of pores and round shape pores have better strength compared to the parts with pore clusters and angular shape pores.

Acknowledgements

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