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Effect of sintering parameters on warm compacted iron-based material ①

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Abstract: Iron-based powder metallurgy material was prepared by warm compaction at 125 ℃ using a compacting pressure of 700 MPa. Sintering temperature ranging from 1 100 ℃ to 1 300 ℃ and sintering time ranging from 40 min to 80 min were used to study the effects of sintering parameters on the compacts. Die wall lubrication polytetrafluoroethylene (PTFE) emulsion was also applied in combination with warm compaction in hope to increase the compact density and the mechanical properties of the sintered material. Green and sintered density, spring back effect and sinter shrinkage were measured. Mechanical properties of both as-sinter and heat treated samples were also measured. Results show that mechanical properties of the sintered compacts increase with the increase of sintering temperature and sintering time. Sample prepared by die wall lubricated warm compaction always shows higher density and mechanical properties.

Key words: warm compaction; powder metallurgy; iron-based material; sintering; die wall lubrication
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1 INTRODUCTION

It is well known that increasing density is the best way to increase the performance of powder metallurgy (P/M) parts. Conventional P/M processing can produce iron-based parts with density less than 7.1 g/cm^3 (a relative density of 90% approximately). Their mechanical properties are substantially less than those of their full density counterpart. There are many methods that can produce P/M parts with relatively high density such as warm compaction, high temperature sintering, double press/double sintering and forging. Warm compaction is a low-cost and simple process with which the high relative density P/M parts can be obtained[1-3]. With minor modification on the conventional equipment and the cost of approximately 20% higher than that of conventional cold compaction, green compact density of 7.5 g/cm^3 can be obtained by single pressing. The only difference between the warm compaction and the conventional compaction is that the powder has to be treated with special lubricant, and then the powder were raised to the prefixed temperature and pressed in the die, which maintained at the warm compaction temperature.

Industrialization of the technique matured in the mid 1990's. The success of warm compaction technique relied on the correct use of special lubricant. However, it is well known that there is a dilemma in using lubricant. On one hand lubricant is essential for metal powder to overcome friction and avoid scoring in the die compaction. On the other hand, the presence of lubricant may lower the sintered density and decrease the mechanical properties of the compact. A mixed lubricant also lowered the flow rate of powder and prolonged the compaction cycle time. Therefore, how to minimize or even to eliminate the use of admixed lubricant in metal powder blends have long been a dream for P/M industrialist.

Admixed lubricant is important when compacting metal powders in a die, since it can reduce inter-particle friction, particle-die wall friction and ejection force. As pointed out by some workers[4], the most important role for lubricant is to overcome the die wall friction rather than inter-particle friction. Several researchers[5-9] have investigated die wall lubrication as an alternative to admixed lubricant in metal powders and some effort have been spent towards systems that can provide die wall lubrication. Ball[9] applied dry powder lubricant on die wall by using a tribostatic spray gun and this system was successfully marketed in 1996[10]. Other authors carried out re-

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searches on die wall lubricated warm compaction[11-14]. Their results showed that the admixed lubricant concentration could be reduced in half.

The aim of this study is to study the effects of sintering parameters and die wall lubrication on warm compaction in hope to obtain a powder metallurgy iron-based material with excellent mechanical properties.

2 EXPERIMENTAL

Chemical compositions (in mass fraction) of the tested material are 1.5%−3.0% Cu, 2.0%−4.0% Ni, 0.4%−1.0% Mo, 0.8%−1.0% C and Fe balanced. The powder was prepared by mixing water atomized iron powder with other alloying elemental powders and a special lubricant. The particle size of iron powder was approximately 147 μm. Carbonyl nickel and Mo with particle size of approximately 5 μm were used. The particle size of Cu was approximately 74 μm. The mixing time was 30 min. Except for die wall lubricated warm compaction, at which 0.25% of the admixed lubricant was used, the admixed lubricant content used in this study was 0.6%.

The pre-heated mixed powders were pressed into standard tensile specimens (ISO 2740-1973) in a steel mold, which was heated to (125±2) ℃. Polytetrafluoroethylene (PTFE) emulsion was brushed on the die wall as die wall lubricant for die wall lubricated warm compaction. A compacting pressure of 700 MPa was loaded. Sintering was carried out at a temperature ranging from 1 100 ℃ to 1 300 ℃ for 40, 50, 60, 70 and 80 min respectively in the sintering chamber of a pusher type furnace, which was protected with a hydrogen-nitrogen atmosphere (from cracked ammonia). The pre-heating and the cooling temperature in the furnace was 700 ℃. The heat-treatment schedule was as follows: annealing at (875±5) ℃ for 60 min followed by quenching in water, then tempering at 200 ℃ for 1 h. Green density, sintered density, ultimate tensile strength (σ), elongation (δ), un-notched impact toughness (a) and apparent hardness were measured. A computer-controlled 100 kN CMT5105 universal testing machine was employed to measure the ultimate tensile strength and elongation at a loading speed of 5 mm/min. Densities were measured by Archimedes method.

Spring-back effect was determined by measuring the difference between the size of the sample and the inner size of the die. The sample size change after ejection was measured by micrometer according to GB/T5159—1985. Size change ratio (Δd_{DG}) is defined as

$$\Delta d_{DG} = [(d_G - d_D)/d_D] \times 100\% \quad (1)$$

where d_G and d_D are the size of the green compact and inner size of the die, respectively.

Sample's sinter shrinkage was measured by micrometer according to GB/T5159—1985. Shrinkage ratio (Δd_{GS}) is defined as

$$\Delta d_{GS} = [(d_S - d_G)/d_G] \times 100\%$$

where d_G and d_S are the size of the green compact and sintered compact, respectively.

Results reported in this study are the average of at least three samples.

3 RESULTS

Fig. 1 shows the relationship between mechanical properties and sintering temperature for both die wall lubricated and non-die wall lubricated warm compacted samples sintered for 50 min. Fig. 1(a) shows the data of tensile strength and elongation, and Fig. 1(b) shows the data of apparent hardness and impact toughness. Except for the elongation data obtained from the samples sintered at 1 100 ℃ and 1 150 ℃, all data increase with increasing sintering temperature. Compared with non-die wall lubricated samples, the die wall lubricated samples have higher mechanical properties.

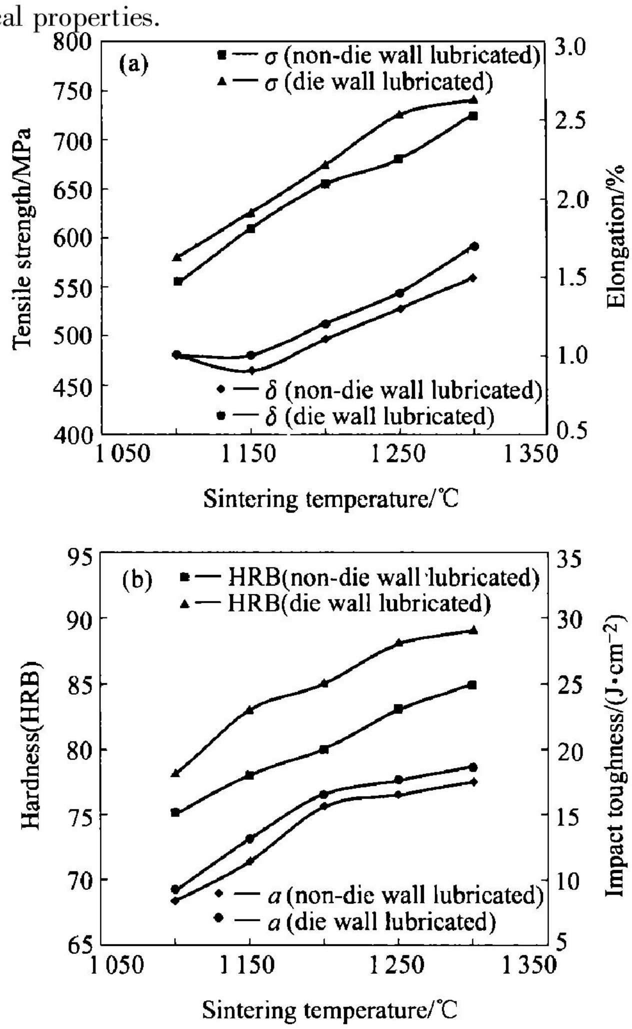

Fig. 1 Plots of tensile strength and elongation (a), apparent hardness and impact toughness (b) vs sintering temperature for samples sintered for 50 min

Fig. 2 shows the relationship between sintered density and sintering time for both die wall lubricated and non-die wall lubricated warm compacted samples sintered at 1 300 ℃. It can be seen that the sintered density increases with the increase of sintering time.

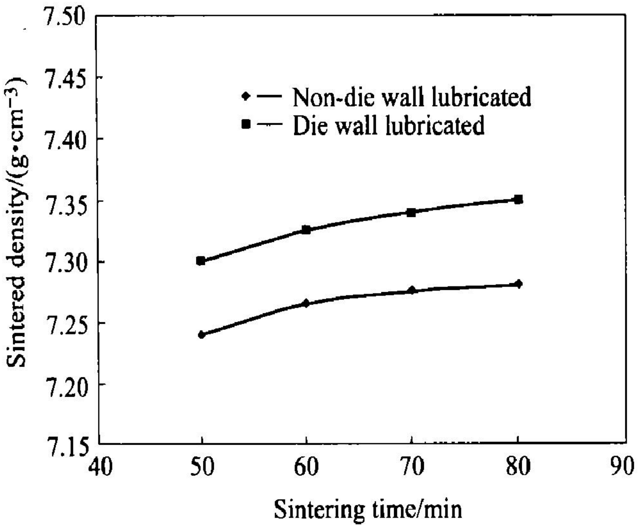

Fig. 2 Relationship between sintered density and sintering time for both die wall lubricated and non-die wall lubricated warm compacted samples sintered at 1 300 ℃

Fig. 3 shows the relationship between mechanical properties and sintering time for both die wall lubricated and non-die wall lubricated warm compacted samples sintered at 1 300 ℃. Fig. 3(a) shows the data of tensile strength and elongation and Fig. 3(b) shows the data of apparent hardness and impact toughness. Except for the impact toughness data obtained from the samples sintered for 60 and 70 min, all data increase with increasing sintering time. Compared with the non-die wall lubricated samples, the die wall lubricated samples have higher mechanical properties.

Table 1 lists experimental results on warm compacted samples sintered at 1 300 ℃ for 80 min. Both density and mechanical properties obtained by die wall lubricated warm compaction have slightly higher values when being compared with those obtained by warm compaction without die wall lubrication. The spring-back effect and the sinter

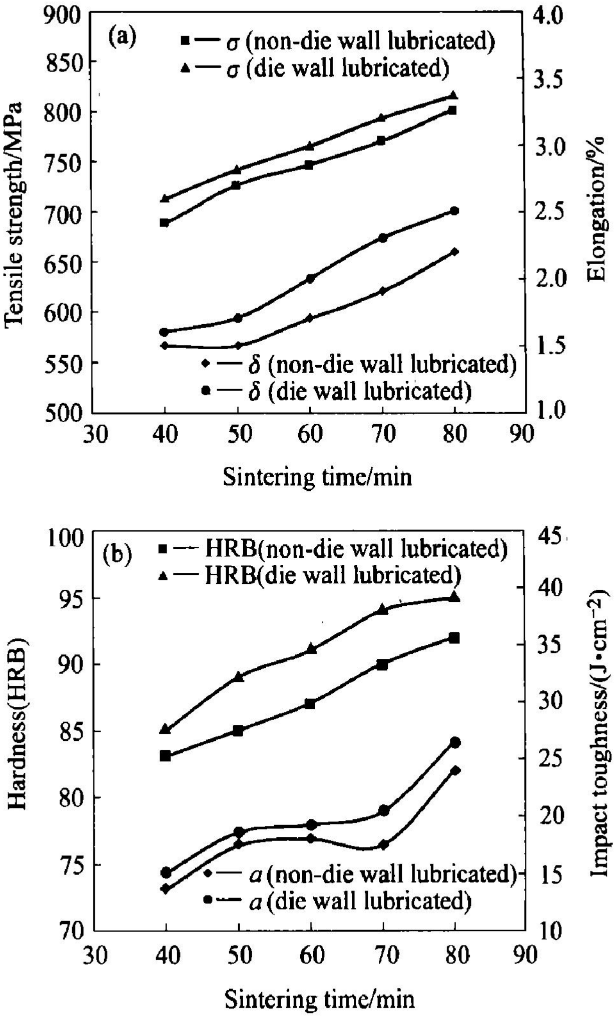

Fig. 3 Plots of tensile strength and elongation(a), apparent hardness and impact toughness(b), vs sintering time for samples sintered at 1 300 ℃

shrinkage for samples obtained by die wall lubricated warm compaction were very close to those obtained by warm compaction without die wall lubrication. The heat-treatment can greatly increase the tensile strength of the samples without sacrificing too much elongation and impact toughness.

4 DISCUSSION

Friction between the compact and the die wall will decrease the effective pressure and increase the ejection force. Die wall lubrication was used to

Table 1 Mechanical properties of warm compacted samples sintered at 1 300 ℃ for 80 min

Die wall lubrication	Green density/ ($g\cdot cm^{-3}$)	Sintered density/ ($g\cdot cm^{-3}$)	Spring back effect/ %	Tensile strength/ MPa	Sinter shrinkage/ %	Elongation/ %	Impact toughness/ ($J\cdot cm^{-3}$)	Hardness
Without	7.31	7.28	0.18	802	0.15	2.2	24	HRB92
With	7.38	7.34	0.18	815	0.18	2.5	27	HRB95
Without (heat-treated)	–	–	–	1 170	–	1.5	21	HRC54
With (heat-treated)	–	–	–	1 210	–	1.5	22	HRC55

maximize the effective pressure. As shown in Figs. 1 – 3 and Table 1, both density and mechanical properties of the samples obtained by die wall lubricated warm compaction were higher than those obtained by non-die wall lubricated warm compaction. Since PTFE has a very low friction coefficient, the major friction problem in compaction occurs between the powder and the die wall. PTFE on the die wall can reduce a great portion of the friction during compaction and thus increase the effective pressure on the powder. Each curve of the mechanical properties for non-die wall lubricated warm compaction and die wall lubricated warm compaction are very similar in nature, which indicates that the compaction mechanism and the sintering mechanism are not affected by the die wall lubrication. The better mechanical properties of warm compacted samples with die wall lubrication is mainly due to the increase in effective pressure and the reduction in admixed lubricant content, which leads to the increase in compact density.

The quality of the P/M alloy depends on the homogeneity of the sintered compact. P/M material inherited an unavoidable disadvantage that the diffusion cannot be completed within the sintering process. The diffusion coefficient of elements strongly depends on the temperature. It is no doubt that higher sintering temperature and longer sintering time can promote the inter-diffusion of elements but the excessively high temperature will lead to the unacceptable size change and even shape deformation. Although the longer sintering time can make an alloy better homogeneity but the problem of grain growth will be encountered instead. Experimental results indicated that sintering temperature of 1 300 ℃ and sintering time of 80 min is an optimum procedure for P/M product with good mechanical properties and dimension stability.

In Fig. 1(a), the overlapping of elongation data for the non-die wall lubricated warm compacted and die wall lubricated warm compacted samples sintered at 1 100 ℃ is believed to be due to the data scattering since for elongation measurement the experimental error are in a range of 0.2%. In Figs. 1(b) and 3(b), the impact toughness curves leveled off after an increase and in Fig. 3(b) the curves increased again after a longer sintering time. The reason for this is not exactly clear but it is believed that the evolution of microstructure is a main factor. Impact toughness is a measurement of the energy absorbed per unit area during the impact fracture, and it related to the strain rate. Unlike tensile test, impact test is a very fast fracture process; and the fracture is always initiated and propagated at the weakest location and there is no chance for the dislocation movement to deviate and absorb the applied energy. It is very sensitive to void concentration and the shape of voids and grains. Microstructure with round grains and round voids can give higher impact toughness since stress concentration at a particular location is not likely to happen and therefore less chances are given for the crack initiation and propagation.

At lower sintering temperature, the inter-diffusion process of elements is very slow. As the temperature increases, the rate of inter-diffusion increases rapidly since the diffusion coefficients of elements increase exponentially with the temperature. For a particular sintering time, as the temperature increases, the inter-diffusion of elements not only leads to the neck growth and the reduction of void concentration but also the formation of large amount of new phases. When these new phases become significant in quantity, they will affect the mechanical properties of the compact, especially the impact toughness. For instance, for relatively low strength steel samples with similar composition and similar tensile strength but different microstructures, the sample with flaky pearlite has a less impact toughness when being compared with the one with bainite[15]. As the quantity of the unfavorable microstructure increases, the impact toughness will be affected even the neck growth and the reduction of void concentration increase the impact toughness. This is the reason why in Fig. 1 (b) that the impact toughness curves leveled off after an increase.

For a given sintering temperature, the sintering process can be divided into several stages[16]: 1) inter-particle bonding, 2) neck growth, 3) closure of pore channels, 4) rounding of pores, 5) pore shrinkage, and 6) pore coarsening. At the initial stage of the sintering, the formation and the growth of necks in the compact will increase the mechanical properties of the compact rapidly. As the sintering time increases, similar to the above argument, the quantity of the unfavorable microstructure will also be increased and thus the impact toughness will be affected even the neck growth increase the impact toughness. The further increase in sintering time will lead to the spheroidization of the voids and homogenization of different phases, which are beneficial to the impact toughness of the sintered compact. This is the reason why we see in Fig. 3(b) that the impact toughness curves leveled off after an increase and then increased again after a longer sintering time.

Higher sintering temperature can produce sample with higher mechanical properties; but as the sintering temperature increases beyond 1 300 ℃, the increase is not so significant. Except for the elongation data, other mechanical property curves of the die wall lubricated sample in Fig. 1 leveled off at 1 250 ℃. Although sintering beyond 80 min may further increases the mechanical properties of the compact, in the viewpoint of cost saving and dimensional stability, sintering parameters of 1 250 ℃ and 80 min are recommended for the tested material. Of course longer sintering time is a possible option.

5 CONCLUSIONS

Both density and mechanical properties of the samples obtained by die wall lubricated warm compaction are higher than those obtained by warm compaction only. The better mechanical properties of warm compacted samples with die wall lubrication are mainly due to the increase in effective pressure and the reduction in admixed lubricant content, which leads to the increase in compact density. Compared with the hardness and the tensile property, the impact toughness of the sintered compact is a strong function of sintering temperature and time. Sintering temperature of 1 300 ℃ and sintering time of 80 min is an optimum procedure for P/M product with good mechanical properties and dimension stability. However, in balancing the production cost and the product quality, sintering at 1 250 ℃ for 80 min is recommended for the tested material.

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