THE INFLUENCE OF THE MORPHOLOGY OF IRON POWDER PARTICLES ON THEIR COMPACTION IN AN AUTOMATIC DIE

Keywords: Fe-based alloy powders, morphology and microstructure of particles, influence on automatic die compaction, sintering

Fe- and steel-based powder metallurgy (P/M) products, such as steel gears, spurs, locking mechanisms, porous filters, sliding bearings and bushes, as well as other machine parts and structural elements, are mainly produced with the so-called conventional sintering technology. It is the most efficient technology for the mass production of small, complex, functional and structural parts. Therefore, it is the most convenient and popular among all of the P/M technologies. The most important end-user of sintered parts is the automotive industry. However, small, complex, sintered parts can also be frequently used in the furniture and household industries, precise mechanics, articles for recreation and sports. A fine, iron-based powder mixture or prealloyed powder is first automatically uniaxial-die compacted (ADC) into the final shape of the product with a mechanical or hydraulic press and then sintered in a protective atmosphere at approximately 1100 °C. The metal powder mixture must have the appropriate engineering properties given by the chemistry and particle morphology, enabling a fast and reliable die-compaction process. The most important are a high tap density, a good powder flowability and a low compressibility. All this gives the green compacts an appropriate final shape with a smooth surface, a relatively high and uniform green density, as well as a green strength without internal flaws and cracks. In the case of very small two- or more-heights products, for example, spur gears with a low module, it is very difficult to obtain a uniform green density at acceptable compaction pressures. Often small cracks are formed at height crossings and big differences in the green density appear in smaller or thinner regions. In the frame of our investigation we analysed the influence of the selected prealloyed commercial iron powder’s morphology and its technological properties on automatic die compaction, as well as the sintering process in the case of small two-level sintered gear dimensions of 5/40–7/10 mm with module $m = 0.5$. The original iron powder was sieved and the finest powder particle fraction (< 45 μm) was compared with the original powder mixture considering ADC and sintering process. It was found that the selection of the finer powder mixture could not contribute to the improvement in the overall ADC process, as well as a better green compact.

In the present paper the results of our investigations are presented and the reasons why a finer powder mixture cannot contribute much to an improvement of the conventional sintering process. The most important are a high tap density, a good powder flowability and a low compressibility. All this gives the green compacts an appropriate final shape with a smooth surface, a relatively high and uniform green density, as well as a green strength without internal flaws and cracks. In the case of very small two- or more-heights products, for example, spur gears with a low module, it is very difficult to obtain a uniform green density at acceptable compaction pressures. Often small cracks are formed at height crossings and big differences in the green density appear in smaller or thinner regions. In the frame of our investigation we analysed the influence of the selected prealloyed commercial iron powder’s morphology and its technological properties on automatic die compaction, as well as the sintering process in the case of small two-level sintered gear dimensions of 5/40–7/10 mm with module $m = 0.5$. The original iron powder was sieved and the finest powder particle fraction (< 45 μm) was compared with the original powder mixture considering ADC and sintering process. It was found that the selection of the finer powder mixture could not contribute to the improvement in the overall ADC process, as well as a better green compact.

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1 INTRODUCTION

The conventional sintering technology is the most popular among all P/M technologies. It enables the large-scale production of small complex parts (Figure 1) with the lowest raw-material and energy consumption.¹⁻³ This technology is used in the Unior forging industry, Sinter workshop, Zreče, Slovenia, mainly for the production of sintered steel parts for the automotive industry.⁴

Sinter technology consists of three main production phases: powder manufacture and the preparation of the mixture, automatic (uniaxial cold) die compaction (ADC) and sintering (Figure 2). The improvement of the dimensional tolerances and the mechanical properties can be obtained with the additional post-sintering operations, i.e., sizing, surface and heat treatment and machining.

Some geometrical limitations exist in the phase of product design and later in the phase of tool manufacture when considering uniaxial automatic die compaction. These limitations have their origin in the nature of uniaxial ADC. Namely, the forces (pressures) of compaction are not uniformly transferred over the height and cross-section of the formed green compact because of the internal friction among the powder particles and the friction on the die walls (Figure 3). This has already been shown by the classic analysis¹ introducing an equation where one can see that the transferred compaction force depends not only on the internal friction and the die-wall friction, but also on the ratio between the height and the diameter of the green compact (h/D):

\[ F_x = F_0 \cdot e^{-\mu z \frac{h}{D}} \]  \hspace{1cm} (1)

In Equation (1) \( F_x \) is the resulting force at a distance \( x \) from the upper punch, \( F_0 \) is the acting (compaction) force on the upper punch, \( \mu \) is the die-wall friction coefficient and the factor \( z \) describes the ratio between...
the normal and the powder-transferred radial stresses, which depends on the internal friction among the powder particles.

From Equation (1) we can calculate the transferred force at any distance $x$ in the formed green powder compact, as well as the lowest transferred force on the bottom punch (at $x = h$) if the die and powder characteristics $\mu$ and $z$ are known. The higher is the ratio $hD$, the larger are the local differences in the green density and the formed green compact has an unequal green density over its volume (Figure 4). The higher is the compaction pressure, the higher is the local and overall (average) green density of the compact.

The green density distribution can be mitigated if the compaction force acts from both sides (top and bottom) when forming the green part. Therefore, modern tools for ADC consist of a large number of parts; their movement is programmed and controlled by a computer on hydraulic or mechanical presses (Figure 5) in order to avoid too large differences in the green density of the compact and its uniform ejection out of the die.

In spite of this, it is not possible to ensure that a green compact has a completely uniform green density over the whole volume (Figure 4), especially, if in the product design phase, it is not possible to avoid larger height differences, sharp crossings and chamfers, because of other functional limitations of the final sintered product. The results of an unsuitable geometry of the product are: non-uniform powder filling of the die, large local compaction pressures, forming large local green-density differences, and finally cracking of the green compact during ejection. However, these also lead to wear/fracture of the most loaded tool parts and overall shorten the life of the tool. Tools (dies) for the compaction of metal powders are very precise, made of advanced tool steels and cemented carbides, and therefore, their manufacture is very complex and expensive. Tool life depends not only on its complexity but also on powder engineering (technological) properties. Metal powders have to have a large tap density, good flowability and compressibility for the appropriate ADC.

One such sintered steel product that has a difficult ADC geometry is the two-height small gear produced in the Unior factory (Figure 6). It also has a very small gear module ($m = 0.5$) as well as large height and diameter differences.
This small gear is compacted with very high compacting pressures in order to decrease the differences in the green density in the gear teeth because of poor filling of the engraving, as well as to decrease the differences in the green density between the gear parts with a large height difference to avoid cracking at the height crossing. This demands high compaction pressures over 700 MPa. The result is a too short tool-life because of frequent fracture of the most loaded tool parts (punches and core rods, Figure 7).

Different solutions (better tool materials, more precise tooling, optimization of the die and press set up) have been researched to solve this problem. But no one has found a complete result and a final solution. Therefore, we also tried with a change of the existing powder granulometry. The hypothesis was that the selection of a finer powder could offer better compressibility and filling of the die. Unfortunately, as it follows, the change of the granulometry to a finer powder also did not give an improvement of the ADC process but gave us a lot of useful and interesting information.

2 EXPERIMENTAL WORK

For the production of the investigated two-height gear a standard commercial diffusion prealloyed Fe-based powder Distaloy AB, Höganäs, Sweden was used. Its average nominal bulk chemical composition in mass fractions (w/%) is: 1.7 Ni, 1.5 Cu, 0.5 Mo, and the rest is Fe. The addition of carbon (generally 0.4-0.6 % graphite) changes it during the sintering into a steel with the required chemical composition. The 5 kg of original Distaloy AB powder was sieved on a set of vibrating sieves in the frame of our experimental work. The finest powder fraction (< 45 μm) was selected for our subsequent experiments and investigations.

The compressibility of the selected fine (< 45 μm) and rough (> 45 μm) mixtures was determined by instrumented apparatus in standardized die dimensions of φ 24 mm × 16 mm. The experiment for the compressibility determination is performed at a ram speed of 10 mm/min. It is a much slower speed than the actual industrial ADC process. Therefore, the densification and deformation rate of the green compact in industrial conditions are different and higher (a larger number of structural defects affecting the sintering), respectively. The flowability and tap density of the selected powder mixtures were determined with a Hall flowmeter. The prescribed amount of graphite (w = 0.5 %) and Kenolube lubricant (w = 0.9 %) were added to the original Distaloy AB and the fine sieved powder and both were then homogenized in a double-cone mixer. The experimental compaction of the gears was performed on an industrial 60 kN Dorst, Germany, mechanical press. Approximately 100 gear pieces were compacted from both powder mixtures followed by the sintering of green compacts in an industrial continuous-belt Mahler furnace under standard sintering conditions (1120 °C/30 min) in a protective atmosphere (N₂ + 5/10 % H₂). The sintered gears were additionally heat treated after sintering (oil quench-
ing from 890 °C and tempering at 200 °C/30 min) The Vickers hardness HV5 of the sintered and heat-treated gears and the mechanical moment (teeth strength) were determined. The local bulk and micro-chemical compositions of the powders, green compacts and sintered gears were determined with an SEM/EDS (Jeol – JSM6500F/Oxford INCA ENERGY 450, INCA X-SIGHT LN2) and an XRF analyzer (Thermo Scientific, Niton XL3t Goldd+).

3 RESULTS AND DISCUSSION

Figure 8 shows scanning electron micrographs of the fine and rough fractions of the investigated powders. The powders do not have large differences in morphology (shape and surface state), with the exception of the particle size. However, micro-chemical SEM/EDS analyses have shown that the local chemical composition of the fine fraction is significantly different compared to the original mixture. The most probable reason is the method of powder alloying. The used Distaloy AB powder is diffusion prealloyed (Figure 9b) and segregation of the alloying elements occurred during sieving and finer powder particles have a different chemical composition than the larger ones. Figure 10 shows EDS micro-chemical mapping analyses of the fine and large powder particles. It is clear that their local compositions are quite different. This was also confirmed by the XRF analyses, which included a much larger volume of analyzed sample. In spite of this, the local chemical compositions of all the samples differ significantly from the nominal chemical composition of the Distaloy AB powder. Table 1 shows the average chemical compositions of all the analyzed samples. It is clear that the fine powder mixtures have a much higher content of alloying elements than the original powder mixture. As will be shown later, this over-alloying also has a significant influence on the mechanical properties of the sintered and heat-treated gears.

Table 2 shows the results of the technological properties of the original, fine and rough powder mixture. We can see from this table that the fine fraction mixture has a poorer flowability, a lower tap density (ρv) and a negligibly better compressibility (ρs at ρu). Our hypothesis was that the finer powder mixture has a better ability to fill the die cavity, but obviously the experiments disprove this.

In this way the original powder mixture has a significantly better flowability, a higher tap density and a negligibly lower compressibility, and is therefore more suitable for ADC. This was also confirmed by our indu-
Industrial experiments of the gear compaction. The powder mixture better filled the die cavity and a higher average green density (approx. 7.0–7.1 g/cm³) of the gears at lower compaction pressures (approx. 180 kN) are obtained. The fine powder mixture did not fill the die cavity so well and a lower average green density (approx. 6.9–7.0 g/cm³) of the gears at higher compaction pressures (approx. 210 kN) were obtained. Besides this, the gears made of the fine powder mixture have poorer mechanical properties after sintering and heat-treatment (Table 3). Figures 11 and 12 show the microstructures of the sintered and heat-treated gears. Figures 11a and 11b show a typical microstructure of a polished sample in the region of the tooth-root of the sintered gear visible under a light microscope (LM). It is clear that the sintered gear made of the original mixture has a larger fraction of large pores, but it is better densified in the gear core. This could be a problem of gear resistance to wear and fatigue. On the other hand, the gear made of the finer powder mixture has well-distributed, finer pores, but it is much less densified.

Figures 12a and 12b show typical microstructures of polished and etched samples of the gears after sintering.

<table>
<thead>
<tr>
<th>Powder type</th>
<th>( \rho_s (\text{g/cm}^3) )</th>
<th>Flowability ( \eta/50 \text{ g} )</th>
<th>( P_{\text{max}} ) MPa</th>
<th>( P_r/\text{MPa} )</th>
<th>( \rho_f (\text{g/cm}^3) )</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Original powder*</td>
<td>3.06</td>
<td>26</td>
<td>7.10 g/cm³ at 600 MPa</td>
<td>5.95</td>
<td>6.94</td>
<td>lubricant Kenolube</td>
</tr>
<tr>
<td>Fine fraction (&lt; 45 μm)</td>
<td>2.75</td>
<td>30</td>
<td>722.6</td>
<td>595.4</td>
<td>7.15</td>
<td>lubricant stearic acid</td>
</tr>
<tr>
<td>Rough fraction (&gt; 45 μm)</td>
<td>2.99</td>
<td>26</td>
<td>738.5</td>
<td>512.4</td>
<td>6.94</td>
<td></td>
</tr>
</tbody>
</table>

* manufacturer’s data
4 CONCLUSIONS

The original prealloyed Fe-based powder Distaloy AB was sieved and the technological properties of the fine and rough powder fractions important for ADC were determined. The fine powder fraction has a lower tap density, worse flowability and a negligibly better compressibility. It was expected that the selection of the fine powder fraction can contribute to an improvement in the ADC process of a small, two-height gear, especially to better filling of the teeth engraving, as well as to a more uniform green-density distribution at a lower compaction pressure. This hypothesis has been disapproved based on experimental and semi-industrial work. It has been found that the selection of the finer powder mixture also has other traps. The sieved finer fraction has a different chemical composition than the original powder mixture. This has an important influence on the sintering and heat-treatment response of the material. Therefore, the poorer mechanical properties of the gears made of the fine fraction were obtained. The open question is also the price of manufacture of the fine powder mixture with the correct chemical composition. For now, the existing ADC procedure for the selected small gears is indicated as optimal. In the future, it will be necessary to find other ways to improve the ADC of small spur gears.

5 REFERENCES

6. B. Šuštaršič et al., An instrumented cell to analyse the behaviour of metal powders during cold uniaxial die compaction, Mater. Tehnol., 35 (2001) 6, 351–360