Lubricant influence on the ejection and roughness of in-die electro sinter forged Ti-discs

Cannella, Emanuele; Nielsen, Chris Valentin

Published in:
Key Engineering Materials

Link to article, DOI:
10.4028/www.scientific.net/KEM.767.171

Publication date:
2018

Document Version
Peer reviewed version

Link back to DTU Orbit

Citation (APA):

General rights
Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

• Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
• You may not further distribute the material or use it for any profit-making activity or commercial gain
• You may freely distribute the URL identifying the publication in the public portal

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.
Lubricant influence on the ejection and roughness of in-die electro sinter forged Ti-discs

Emanuele Cannella1, 2, a * and Chris Valentin Nielsen2, b

1IPU Innovation Factory, Produktionstorvet 425, Kgs. Lyngby 2800, Denmark
2Technical University of Denmark (DTU), Produktionstorvet 425, Kgs. Lyngby 2800, Denmark
aemcann@ipu.dk, bcvni@mek.dtu.dk

Keywords: electro sinter forging; titanium; lubrication; ejection; roughness.

Abstract. Electro Sinter Forging (ESF) is a new sintering process based on Joule heating by high electrical current flowing through compacted metal powder under mechanical pressure. The whole process takes about three seconds and is based on a closed-die setup, where the sample is sintered inside a die. A near-net shape component is therefore manufactured. One of the challenges associated with this process is the ejection of the sample after sintering. Due to powder compaction and axial loading during sintering, a radial pressure is generated at the die/sample interface. Consequently, the ejection can be difficult, and the final quality of the sintered component in terms of roughness and surface defects may be affected. In the present work, four different lubricants and non-lubricated conditions were tested to investigate the effects on the final part quality. The sintered sample is a disc made of commercially pure titanium powder. The force was measured while ejecting the samples by using a speed-controlled press. The surface roughness parameter Sa was measured by using a laser confocal microscope.

Introduction

Generally, a conventional sintering process involves metal or ceramic powder [1]. A closed die setup is used to compact the powder, which is therefore ejected as a powder compact, called the green body. The green body is brought into an oven with a temperature, which has to be lower than the melting temperature of the part material. The part is sintered by keeping the compacted part in the oven for a certain time. The particles reduce the free-surface energy and bond together. New sintering processes have been developed during the last decades. The new technologies are focused on the reduction of the sintering time and effects connected to grain growth because of the long sintering time, which is about 30 minutes and depending on the material. To achieve this, an electrical current is used to heat the sample by Joule heating [2]. Electro Current Assisted Sintering (ECAS) comprises all the processes based on this principle [3]. The main difference with the conventional sintering is that the two main phases, compaction and heating, are both carried out inside the die. The powder is firstly compacted to the green body and therefore sintered. The applied punch pressure is kept during the whole process, as in sintering under pressure. When the sample is ejected, it is a near net-shape component. The specific electrical assisted sintering process investigated in this paper is Electro Sinter Forging (ESF) [4].

The problems related to compaction and ejection of the samples are the same in conventional and electrical assisted sintering. Both the green body from a conventional sintering and the sintered sample from an electrical assisted sintering process are subject to the friction along the die-wall. A number of factors influence the friction, e.g. lubrication, compaction pressure, roughness of the tools and friction between particles [5]. The compaction pressure generates a radial pressure on the die-wall during the process. This makes the ejection of the component difficult. The radial pressure can be estimated as 0.46 of the compaction pressure [6]. As demonstrated by Roure et al. [7], the 0.46 value is constant throughout the compaction. Lubrication is well-known in forming as one of the most influencing variables. Generally, in sintering, lubricants are used to improve the ejection
of the samples [8]. The formation of the lubricant film is strictly dependent on the roughness and adhesion of the surface [9].

This paper is focused on the evaluation of the effect of different lubricants on the resulting ejection force and achieved surface roughness. The tested lubricants are powder and spray based. The comparison includes also the non-lubricated case as reference.

Admixed and die-wall lubrication

Two different lubrication methods are described in literature, namely admixed and die-wall lubrication [5]. In admixed lubrication, the powder is mixed with the lubricant, while the die-wall lubrication is based on the application of the lubricant directly on the die-wall. Admixed lubrication is commonly used in industry, where there is the need of an economic and fast process to lubricate the sample. However, the main problem with the admixed powder is on the affected final density and mechanical properties [10]. Lubricant can be trapped and not able to come out from the sample. In conventional sintering, debinding is made before the sintering itself, to allow the binder/lubricant to evaporate. In electrical assisted sintering, this is not possible because of the simultaneous compaction and sintering. Die-wall lubrication was investigated in this work.

Case study

The sintered component was a disc-shape sample with 10.3 mm diameter and 2.5 mm thickness as shown in Figure 1. The disc was made from commercially pure titanium powder, grade 2 (GoodFellow, England). The particle size was 150 µm with purity 99.5%. The electro sinter forging was carried out in an electrical resistance welding machine by Expert Maschinenbau GmbH, Germany, Figure 2, which is mechanically operated by hydraulics and disc springs for follow-up of the force. The electrical control unit is by Harms+Wende GmbH, Germany. The discs were in-die manufactured according to the ESF principle consisting of compaction of the powder and the delivery of electrical current under the applied compaction pressure. Die and electrodes related to the tool setup are shown in Figure 3 and Figure 4. The die is made from an alumina core, to make it electrically insulated, and surrounded by a steel ring to give strength. The electrical current and the load during the process were monitored by using a Rogowski coil and a load cell.

Figure 1: Sintered titanium disc.

Figure 2: Electrical resistance welding machine used for sintering experiments.
The nominal, experimental parameters listed in Table 1, namely compacting pressure, sintering time and electrical current density, were the same for all the experiments. However, the measured process parameters showed some deviations from the nominal values as exemplified in Figure 5 and Figure 6. This is because of the dynamic material properties of the sample during sintering. The tested lubricants are listed in Table 2. In addition, dry conditions were tested. Die-wall lubrication was applied by using a brush for the powder-based lubricants. Molykote D 321-R was sprayed as an anti-friction coating. The lubricants were homogeneously applied to cover the whole internal surface, and the thickness was depending on the particle size and sprayed coating. Five repetitions were made with all lubricants. The ejection force versus ejection stroke was measured for each sample during ejection as shown in Figure 7.

**Table 1: Process parameters for the experiments.**

<table>
<thead>
<tr>
<th>Compacting pressure</th>
<th>Sintering time</th>
<th>Electrical current density</th>
</tr>
</thead>
<tbody>
<tr>
<td>72 MPa (6 kN)</td>
<td>150 ms</td>
<td>96 A/mm² (8 kA)</td>
</tr>
</tbody>
</table>

**Table 2: List of the used lubricants, their composition and particle size/layer.**

<table>
<thead>
<tr>
<th>Lubricant</th>
<th>Composition</th>
<th>Particle size [µm]</th>
<th>Layer [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zinc stearate</td>
<td>ZnO 12.5-14%</td>
<td>44</td>
<td></td>
</tr>
<tr>
<td>Lithium stearate</td>
<td></td>
<td>44</td>
<td></td>
</tr>
<tr>
<td>Acrawax C</td>
<td>Ethylene distearamide</td>
<td>10</td>
<td></td>
</tr>
</tbody>
</table>
The lateral surface roughness of the sintered samples was analysed with a laser confocal microscope (Olympus, Japan). The surfaces were characterised in terms of Sa. The obtained values were compared to the die roughness.

Experimental results

Two main conditions can be described in an ejection load diagram: static friction to initiate movement of the sample and subsequent dynamic friction. The results showed how the use of both powder and spray lubricants helped in reducing the force and the total energy required for ejection as compared to dry conditions. The average ejection forces over the 25 mm ejection stroke shown in Figure 8 and Figure 9 were calculated for each lubricated condition. A number of factors influenced the deviations between samples with the same lubricant. These are 1) the irregular roughness of the die, 2) the compaction randomness between the particles [9], and 3) heterogeneous adherence of the lubricants to the die. Table 3 lists the total energy needed for ejection, which is calculated by integration of the ejection load curves.

Zinc stearate proved most effective in reducing the ejection force, while lithium stearate was the second best. Molykote D 321-R and Acrawax C gave similar results in terms of ejection energy, but the Molykote D 321-R resulted in a larger ejection force at the static ejection and in a larger deviation of the measured values. Furthermore, application of Molykote D 321-R colours both the
die and the disc black. Powder lubricants are easier to remove from the samples. As expected, dry conditions resulted in the highest ejection load. The disc was broken in several experiments and part of the sample was stripped and remained adherent to the die-wall as shown in Figure 10.

Figure 8: Comparison between average ejection forces versus ejection stroke for the different lubricants.

Figure 9: Detail of the static friction for the ejection forces for different lubricants shown in Figure 8.
Table 3: Measured ejection energy for each lubricant over the total ejection stroke of 25 mm.

<table>
<thead>
<tr>
<th>Lubricant</th>
<th>Energy [J]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zinc stearate</td>
<td>2.41</td>
</tr>
<tr>
<td>Lithium stearate</td>
<td>3.62</td>
</tr>
<tr>
<td>Acrawax C</td>
<td>6.31</td>
</tr>
<tr>
<td>Molykote D 321-R</td>
<td>6.11</td>
</tr>
<tr>
<td>Dry conditions</td>
<td>8.73</td>
</tr>
</tbody>
</table>

Figure 10: Sintered material stuck on the die wall after ejection of the sintered disc.

One sample for each lubricant was analysed by using a measuring laser microscope with x50 lens. The discs were cleaned with an ethanol solution before measuring. Two measures were taken at four equi-distant locations at the lateral surface. Figure 11 shows an example of a surface acquired by an analytical software for microscopy (SPIP, Denmark), which was also used to calculate Sa for each surface. The alumina die was previously measured to have an indication of the smallest roughness value achievable. The obtained values for the discs resulted higher than the one measured from the die. Figure 12 gives an overview of the measured surface roughness. The reason for the samples to have larger roughness than the die is the pore nature of the sintered components, which showed large porosity at the surfaces. The large porosity increased the Sa value for all the samples. Regarding the lubricant effects on Sa, it was highlighted how the Acrawax C had a better effect if compared to the other lubricants. However, the standard deviation is large for all the samples. Dry conditions had the largest standard deviation.
Figure 11: Example of one of the acquired surface topographies (Acrawax C).

Figure 12: Results concerning the surface roughness measurements.

Visual comparison, Figure 13, of the surfaces show how the samples made with the zinc stearate had the highest quality. The larger ejection force when using other lubricants produced some stripping effect on the discs, which damaged the original disc-shape. Furthermore, Molykote D 321-R has the drawback of additional cleaning needed as compared to the powder-based lubricants.
Conclusions

The effects of five different lubricated conditions on electro sinter forged discs have been shown. The lubricants were applied as die-wall lubricants, i.e. without mixing them to the powder. Die-wall lubrication gives better final density of the sintered sample than the admixed lubrication. Zinc stearate, lithium stearate, Acrawax C and Molykote D 321-R were tested and compared to dry conditions. To test the lubricant efficiency, the force was measured during the ejection of the samples from the die. Application of zinc stearate resulted in the lowest ejection force, while Molykote D 321-R was the least effective and resulted in the largest deviation of the measured values. Additional characterisation was based on measurement of the surface roughness. The results showed how the pore nature of the sintered samples increased the final roughness as compared to the die roughness. Furthermore, a large standard deviation in the roughness was observed for all the lubricants. The visual observation of the lateral surfaces highlighted the stripping effect during ejection, which influenced the final disc-shape. The samples sintered with zinc and lithium stearate showed better final shape.

Acknowledgments

This research work was undertaken in the context of MICROMAN project (“Process Fingerprint for Zerodeflect Net-shape MICROMANufacturing”, http://www.microman.mek.dtu.dk/). MICROMAN is a European Training Network supported by Horizon 2020, the EU Framework Programme for Research and Innovation (Project ID: 674801).

References


