EFFECT OF GREEN DENSIFICATION MODE ON FRACTURE SURFACE MICROMECHANISMS IN LOW ALLOYED MOLYBDENUM SINTERED STEELS

M. Campos, L. Blanco, J.M. Torralba

Abstract
High density low alloyed PM steels are nowadays a must for high performance applications. In this research, the densification is mainly promoted in the compaction stage and afterwards during sintering. This enables the achievement of higher static and dynamic mechanical properties. Compaction and sintering stages determine the performance of the parts, so the parameters of both have to be carefully chosen and controlled. The investigation of the change that is induced due to high green densities, in terms of pore morphology, fracture micromechanisms and eventually distribution of the present phases, can effectively assist to understand the improvement of the final properties of the materials. In this way, low-alloyed high performance materials with green densities of 7.1 g/cm³, 7.4 g/cm³ and 7.6 g/cm³, have been investigated in as sintered state.

Keywords: high green density, pore morphology, sintering, fracture

INTRODUCTION
It is necessary to accept new challenges to develop new sintered products by using new processes, keeping, at same time, a higher level of strength and tolerances with a low cost. The problem can be approached either by modifying material composition or the pore system. The optimum materials composition complies with low cost alloying elements and with bonding technology to enhance high strength performance and improved tolerances. One advantage of increasing the density during the compaction is the improvement of dimensional control, since the sinter shrinkage is decreased.

The pore system determines the as-sintered properties and one of the most important parameters, beside pore shape and morphology, is the total porosity because it reveals whether pores are mainly isolated or interconnected. Hence, pore characteristics define the behaviour of sintered materials with the same composition [1-3].

High density low alloyed steels can improve dynamic properties, opening up new opportunities to use PM in high performance applications [4-9]. The densification or determination of the pore system can take place at different processing stages; during compaction, sintering or finishing operations or designing high compressibility powders besides the typical double pressing double sintering [10]. If the densification improvement is done during compaction, among others techniques, the die wall lubrication reduces the admixed lubricant amount, and can increase the density up to 7.2 g/cm³. Warm compactions or the high velocity compaction can achieve higher density level, but are not suitable for multilevel parts.

High velocity compaction or cold forging are the new techniques under development that have the potential to bring in new applications for sintered parts. The
process can compact large parts up to a mass more than 5 kg. Powder is compacted in less than 20 ms by high-energy impact. Further densification is possible by adding multiple impacts as short 300 ms after each other.

In this paper an attempt is made to establish a correlation between fractures at sintering necks with the characteristics of pore system in the as sintered state, regarding the green density determined by the compaction technique. Pores are statistically quantified as described in [11,12] and are correlated with the micro mechanism of fracture since free surfaces decrease and the ability of the material to plastically deform changes as a change from interconnected to isolated porosity is produced [13].

EXPERIMENTAL DEVELOPMENT

The materials used in this study were chosen from commercial powder grades (production Höganäs AB) with the same graphite addition as UF4 (Table 1). Besides the green density, the level influence of Mo amount is evaluated as well.

Table 1. Materials used within this paper.

<p>| Added Carbon content: 0.6% C for all materials |</p>
<table>
<thead>
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<th>Nomenclature</th>
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<td>Fe-0.85Mo</td>
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<td>Fe-0.85Mo-2Ni</td>
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<td>Fe-1.5Mo</td>
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Tensile test pieces, as described ISO 2740; were pressed either by normal compaction (NC, double punch automatic press), to produce materials with a density of around 7.1 g/cm³, or by HVC (High Velocity Compaction, shock wave compaction with a hydraulic hammer [14]), to obtain densities around 7.4 and 7.6 g/cm³. Samples were admixed with the same amount of Acrawax.

Specimens were sintered at 1120ºC for 30 min in a 90N2-10H2 protective atmosphere, in a laboratory belt furnace. In this way, the three alloying systems are going to be evaluated on the basis of pore characteristics, such as average pore area, average maximum length and roundness of pores (by F_circle), and their influence on the detected fracture micromechanisms after tensile testing of specimens with different density levels properties and their influence on fracture micro mechanisms detected depending on the mentioned green densities levels.

Basic pore characteristics assessed by image analysis, were studied statistically as described in [15]. Sample preparation was carefully done, opening pores at the polished surface since the density measured by image analysis agrees with the value obtained by the Archimedes’ method. The number of pores, the adjustment of image definition to reveal the pores, was settled with previous work and as referred in [16,17]. The pore system was characterized by pore area, roundness and maximum length (as Feret’s diameter). With this collected information, micro mechanisms of failure at broken surfaces of tensile specimen are discussed.

RESULTS

Figure 1 shows the evolution of particle morphology from green compact with the three levels of density. To make visible the larger interparticle contacts after HVC, samples were prepared taking the die wall face of the specimens, following the pressing direction. Besides changes in the contact between particles, it is noticeable that for 7.4 and 7.6 g/cm³
materials that the particles are certainly flattened in the compacting direction, compared with 7.1 g/cm$^3$ which show particle morphology with low deformation in the same direction.

<table>
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<tr>
<th>a) 7.1 g/cm$^3$</th>
<th>b) 7.4 g/cm$^3$</th>
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<tr>
<th>a) 7.6 g/cm$^3$, surface</th>
<th>a) 7.6 g/cm$^3$, centre of the specimen</th>
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Fig.1. Images of green compacts showing the particle deformation depending on green densities. Unetched.

It would be more difficult to state whether the particles are more deformed at 7.6 g/cm$^3$ than at 7.4 g/cm$^3$. But there is no doubt in that the shape of the particles (more rounded in NC material, and more elongated in HVC) is maintained along all the compacting direction, this points out that the densification level is the same in this direction for these kinds of specimens, i.e., there are not differences between the face of the upper punch and the face of the lower punch. Therefore, the compaction method modifies the pores in terms of amount, size and morphology, at same time provides wider particle contacts. Both factors will contribute to improve the mechanical response because there is an improvement in the load transfer mechanism. Here is considered the ability to transfer the external load through the particle, and how it changes with the green densification level.

High green densities result in a decrease of shrinkage regarding to the material conventionally compacted (NC, green densities 7.1 g/cm$^3$), i.e., dimensional change decreases for materials with a high green density. During sintering, mass transport does not contribute significantly to widen the necks, and decreasing the pores, mass transport essentially concerns self-diffusion between particles, to achieve chemical bonding, and to homogenize the alloying elements. These materials show the enhancement as expected compared to considering NC sample steels as reference, Fig.2.
Fig. 2. Percentage of Dimensional change decreasing, with reference to the value for 7.1 g/cm³.

By plotting pore characteristics vs. UTS is possible to detect the influence of Ni additions and the influence of green density on pore characteristics, Fig. 3. Pore area, roundness (by $F_{\text{circle}}$) and pore length have been measured by image analysis; the obtained values have been analysed with the aid of statistical techniques where mean (average) and standard deviation values have been obtained for each feature of porosity and each material, so the average value has a 95% probability to be within the confidence limit. This confidence interval is lower for materials pressed up to 7.4 g/cm³, they presented a low spread of values. Besides, pores are smaller and have more roundness than 7.1 g/cm³ steels. For the same level of density, Ni additions contribute to round the pores and to decrease their area and decrease the confidence gap. Therefore, the increase in UTS is not only due to Ni presence but also to pore modification.

Fig. 3. Pore characteristics and UTS of sintered materials.

Since necks between particles are the weakest points in as sintered steels; their area and extension determines the consequences of the external load. The ultimate effect leads to material failure; in a microscopic range, the failure is located at particle necks or through the particles [18,19]. The most unfavourable fracture is when necks break by cleavage; because this involves the minimum energy absorption by local deformation. Signs of ductile fracture, such as voids, imply that the materials have been able to consume...
more energy and transmit more external load before failure. If necks can transmit enough load, fracture can be located across the particle, across their grains, transgranular or transparticle, this is the mode to consume more energy before failure [20].

Even though material composition does not lead to a brittle behaviour, necks can fracture with a brittle pattern if pores concentrate and multiply the external stresses to a one-magnitude order higher. Consequently there is a connection between pore morphology and area of particle contacts. As pores become more irregular, with a low $F_{\text{circle}}$ value, the tips of those pores concentrate the stress and can produce a fast local yielding, giving the cleavage.

Fe-0.85 Mo

Density of 7.1 g/cm³.

Fe-0.85Mo+2Ni

Density of 7.4 g/cm³.

Density of 7.6 g/cm³.

Fig.4. Fractures belonging to the centre of the sample.
In general, as ductile signs decrease on the fracture surface, the brittle ones increase with the green densification, Fig.4. Normal compacted materials have shown a great amount of pore surfaces with dimples and micro voids at sintered necks. As the pore amount decreases, by means of HVC, fracture mechanisms change compared to the former density level. Cleavage patterns are more frequent in a higher density system and they are placed across the particle.

Although nickel provides some Ni-rich austenite areas that can transform to martensite by strain-induced transformation under external load, increasing the cleavage areas [21], this phenomenon only takes place easily when steels have higher carbon contents. The Nickel content, of this family of sintered steels, does not modify the micro mechanism of fracture; presence of transgranular brittle patterns is very similar in both materials as well as ductile signs, in this study is shown more strongly the effect of density than the Ni-alloying.

This idea is to some extent reflected on yield strength values, Fig.5, the yielding of the sintered steels is held up with the increasing of green density and with the Ni additions. Although in this case, is more effective the addition of Ni to raise this property, only considering the synergetic effect of Nickel and high density is possible to overcome 500 MPa.

![Fig. 5 Yield Strength of the materials.](image)

For the 7.1 g/cm$^3$ steels family, the yield strength is in accordance to the bainitic microstructure with that carbon level, Fig.6. By increasing the density up to 7.4 g/cm$^3$, sintered Ast85Mo steels can achieve similar yield strength as those obtained by prealloying with 1.5Mo content (Astaloy Mo). By comparing with the Fe-0.85Mo-2Ni steels, it is evident the improvement is provided by austenite Ni-rich grains at this carbon level.
CONCLUSIONS

Green densification provides materials with a large contact between particles and allows for controlling better the tolerances of the compacts, minimizing the shrinkage during sintering. Materials with high green densities achieve a high performance; green densities change the governing fracture mode and contribute to enhance the material response.

The amount of pores and their morphology characteristics modify the load transfer mechanism because it modifies the degree of raising the external load. The transgranular failure produced through the particles for higher green densities is more positive than ductile fracture only in particle necks.

Concerning the low alloyed Molybdenum steels, the amount of alloying element could be reduced if it achieved higher values of density (≥7.4 g/cm³).

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REFERENCES


