STUDY OF THE EFFECT OF THE PROCESSES PARAMETERS ON POROSITY OF LOW ALLOYED SINTERED STEELS BY IMAGE ANALYSIS

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Dedicated to Dr. Andrej Šalak at the occasion of his 80th birthday.

Abstract

Composition and pore system are two important events that determine the sintered material response. The purpose of this study is to investigate the effect of processes parameters on pore features and in particular the bigger pores, since these bigger pores are directly responsible for the mechanical properties (especially the dynamic ones) that a material shows. In this way, by means of image analysis techniques, the porosity study is conducted to study the effect of the 10 % bigger pores and the effect of the whole pore system obtained after achieving green densities of 7.1 and 7.4 g/cm³. So pore area, maximum length and roundness are analysed for the two densities and for two sintering temperatures, as well as the effect of thermo chemical treatments like the carbonitriding. Results are correlated with mechanical properties.

Keywords: image analysis, pore features, high sintering temperature, Mo-Ni low alloyed steels

INTRODUCTION

Porosity is a very important characteristic of sintered components that affects the properties [1-4]. The most important parameters are total porosity and shape of pores/sintering contacts. Depending on total porosity which is influenced by the compacting pressure –and the sintering conditions, the sintering contacts can be isolated or interconnected [2]. Not just the percentage of pores, but the size of those pores and their shape, together with their distribution within the structure and the distance between them can picture in an indirect way the final behaviour of the material, these factors can be controlled by careful adjustment of the processing parameters [5].

Nowadays, one of the major interests of the PM industry is to achieve high performance through high densities. With this aim, highly compressible powders, and innovative pressing methods like warm compaction or "high velocity compaction" (HVC) have been developed to make possible the manufacturing of high-density components without using double pressing and double sintering [6]. A higher densification gives higher fatigue properties and broadens the material applications [7-12].

In this paper we intend to establish a proper method in order to quantify pore features statistically, i.e pore area, pore maximum length (Feret's diameter) and pore roundness (F factor), and to compare populations of these features among them [13, 14]. This comparison is made for as sintered materials (using two different sintering conditions, i.e. lab scale and large scale) and for as sintered and heat treated materials. The

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methodology has been applied also to the study of the difference in area, maximum length and roundness that the top 10 % coarser pores possess, since these bigger pores are generally the most damaging for the PM components.

EXPERIMENTAL DEVELOPMENT

The materials used, have been developed from commercial powder grades, produced by Höganäs AB (Sweden). The description is given in Table1. The effect of chemical composition is considered by comparing the two materials, since Astaloy 85 Mo was blended with nickel powder (2 %), providing Ni-rich areas, and modifying the diffusion progression. Carbon was added as UF4 graphite in the mixing step.

Tab.1. Materials and processing parameters used within this paper.

Nomenclature	Features	Processing Conditions	
		Compaction	Sintering
Astaloy85Mo+0.6%C	Water atomised iron powder, prealloyed with 0.85% Mo	NC &HVC	1120ºC &1250ºC
Astaloy85Mo+2%Ni+0.6%C	Mechanically blended with 2% of Ni powder	NC &HVC	1120ºC &1250ºC

Tensile test bars were pressed under two different conditions, attaining two density levels: 7.1 and 7.4 g/cm^3. By means of uniaxial compaction (double punch automatic press $\cong$ Normal Compaction, NC), 7.1 g/cm^3 green density; and by means of HVC, (high velocity compaction, shock wave compaction with a hydraulic hammer [15]) to obtain green densities around 7.4 g/cm³ Acrawax was used as a lubricant keeping the same proportion due to its importance on pore features. Specimens were sintered at two different conditions following the same cooling rates, between 0.5-1ºC/s; lab scale sintering conditions -1120ºC for 30 minutes in $90N_2$-$10H_2$, laboratory belt furnace- and lab scale sintering conditions -1250ºC for 60 minutes in $90N_2$-$10H_2$, laboratory belt furnace. Thus, two different compaction methods, and two different sintering conditions are going to be evaluated on the basis of pore features, such as average pore area, average maximum length and roundness.

The effect of the surface treatments on pore features is studied by comparing sintered conditions for both densities vs. the same material families after carbonitriding in industrial conditions -sintering at 1100ºC/45 min. in N_2+DA followed by carbonitriding, austenitizing at 850ºC with a carbon potential of 0.6% during 60 min., then oil quenching at 70ºC.

In order to get enough populations of pore area, length and roundness to be statistically analysed afterwards, pieces of the tensile samples were mounted and polished for metallographic examination and image analysis. One thing of major importance regarding quantitative image analysis, is the specimen preparation, the brightness and the contrast used to discriminate features, number of fields examined, total surface examined and image magnification (in this case settled at 20x based on preliminary experience with the analysis software used for this research) [16]. The samples were polished in order to get the same density values when measuring by the Archimedes method and when measuring by image analysis. After that, image analysis is run through the polished area, taking a total of 50 pictures of the whole surface, and quantifying not less than $2.0 \cdot 10^4$ pores, to quantify pores varies from a minimum of $2.3 \cdot 10^4$ to a maximum of $3.5 \cdot 10^4$. Pore area, maximum length and roundness measurements are obtained from the total number of pores. Maximum

length was calculated as Feret's diameter, the measured distance between parallel lines that are tangent to the object's profile and perpendicular to the ocular scale. Pore roundness was calculated according to [17] (eq. 1).

$$Roundness = \frac{4 \cdot \pi \cdot Area}{(Perimeter)^2}$$ (1)

When roundness is equal to 1, the pore tends to a circular shape, if roundness is close to zero, the pore looks like a line. Once image analysis techniques were applied, the information was analysed by means of statistical techniques, using the software Statgraphics Plus 5.0. Then mean value (average) and standard deviation values were obtained for each feature of porosity and each material. These values are analysed in an interval form for a 95 % confidence level (Fig.1). With these values, it is possible to compare in pairs lists of values.
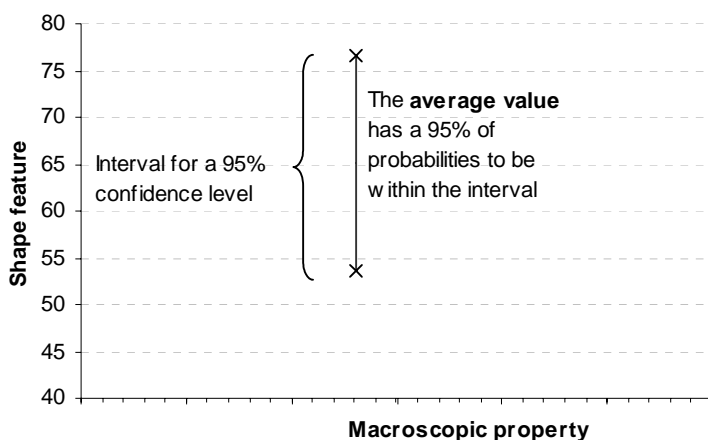
Fig.1. Example of the meaning of an interval for a 95 % confidence level.

RESULTS AND DISCUSSION

It is difficult to picture the direct influence of porosity parameters on tensile properties, since we must also take into account the effect of microstructure like phase distribution. Other effects such as lubricant burnt can be neglected in this case, because for all materials, the same type and amount of lubricant was used. After the metallographic discussion is considered, pore features and the effect on UTS will be examined.

Metallography features

In Figure 2, the microstructures obtained for [Astaloy85Mo+0.6C] and [Astaloy85Mo+2Ni+0.6C] are shown. For the former, upper bainite was obtained as the dominant constituent, while a mixture of upper bainite, Ni-rich retained austenite and martensite were obtained for the Ni-alloyed. In terms of metallography, regarding sintering temperature there are only differences in Ni-alloyed steel, where high temperature sintering decreases the Ni-rich areas as the diffusion process is enhanced.
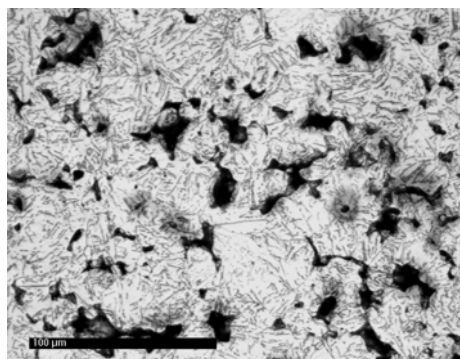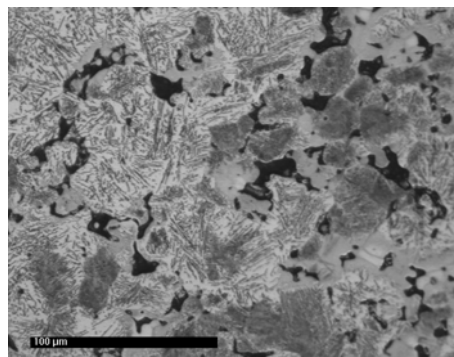
Ast85 Mo, 1120ºC sintered
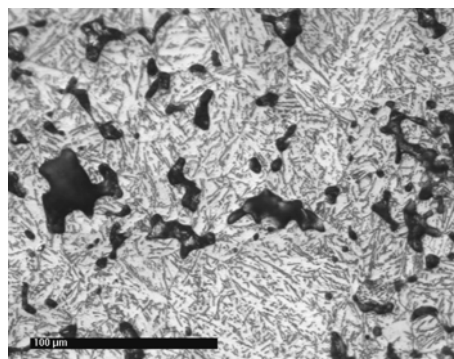
Ast85 Mo, 1250ºC sintered
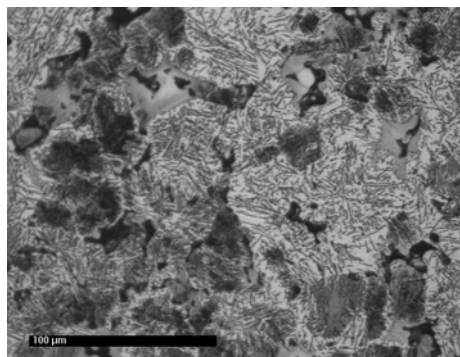
Ast85 Mo+2Ni, 1120ºC sintered

Ast85 Mo+2Ni, 1250ºC sintered

Fig.2. Microstructures of the two materials.

Pore analysis

NC and 1120ºC sintering temperature can be considered as "conventional" processing parameters. Hence the fact that an improvement of the material response can be found by increasing green densities by HVC, by increasing densification raising the sintering temperature up to 1250ºC or by both methods at same processing route. In Figure 3, the HVC results to smaller pore areas, shifting the values to the left part of the graphic, and 1250ºC moves the values in a right-top direction when green densities are low, i.e. NC, which implies rounded pores but slightly bigger. This result is in accordance with [18]. When the sintering of materials starts from high green densities (HVC), the pores do not decrease the area significantly.

Considering the 10 % of bigger pores (Fig.4), the results highlight the influence of HVC and 1250ºC sintering temperature, because the bigger pores are the ones which suffer the higher transformation. HVC involves a higher densification than 1250ºC sintering temperature since higher values of pore area are achieved. However, the bigger pores are smaller when densification is attained by a 1250ºC sintering temperature, if material is processed by HVC and high sintering temperature, the densification is read as a lower pore area, the smallest pores and the highest roundness. In view of the densification achieved by HVC or high temperature sintering, it is suggested that pore area is mainly determined from the compaction step [19], whereas pore roundness is mainly determined at the sintering stage.
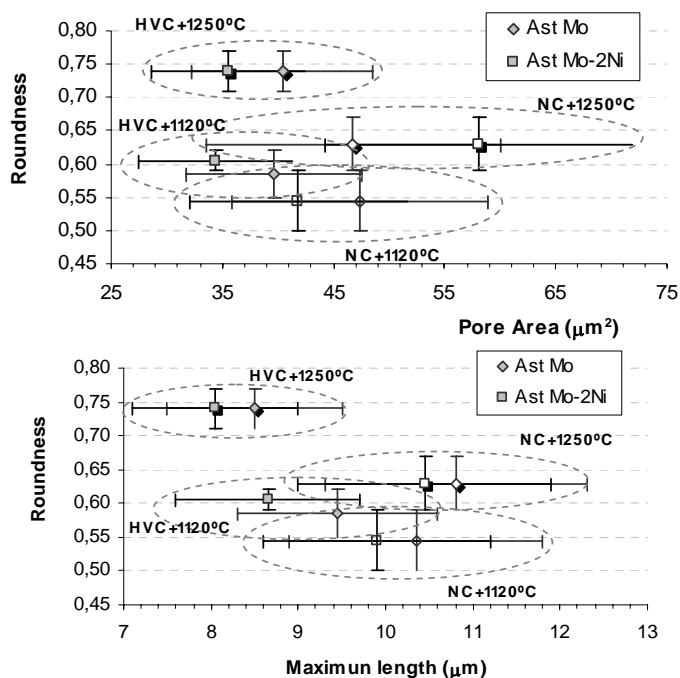
Fig.3. Roundness vs Pore area or Maximum length for both materials processing by NC or HVC and sintering at 1120º and 1250ºC.
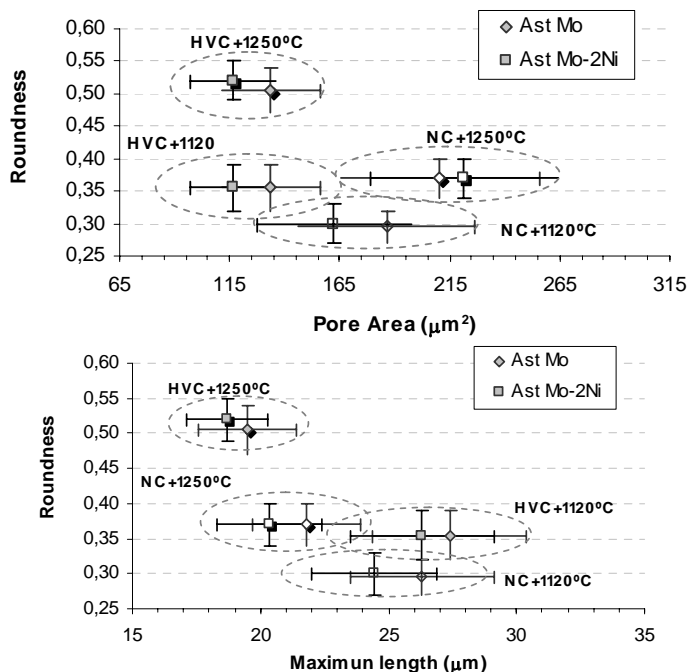

Fig.4. Roundness vs. Pore area or Maximum length for both materials processing by NC or HVC and sintering at 1120º and 1250ºC considering the 10 % of bigger pores.

Comparing Figure 3 with Figure 4 the conclusion is that by alloying with nickel, lower values of pore area, and pore maximum length, are obtained and as a result higher tensile strength are achieved Fig.5. This effect is much more visible analysing just the coarser pores. These materials with nickel behave better from the measured static properties since nickel, apart from reducing the pore area and length in these materials, also strengthens the matrix like when it is used as alloying element in wrought steels. However, the addition of nickel also results in a microstructure consisting of retained austenite which has a positive effect on the dynamic properties [6] but this is not to be discussed in this paper. Ni alloyed materials show slightly smaller pore areas than materials without this element (especially when Ni is blended, in Astaloy85Mo+2Ni), this is a consequence of the shrinkage effect of Ni in sintered materials [20].

In Figure 5 is shown the relation between roundness and UTS, this relation is notice taking into account the whole pore system or only the 10 % of bigger pores. As sintered material have more rounded pores, the UTS increases.
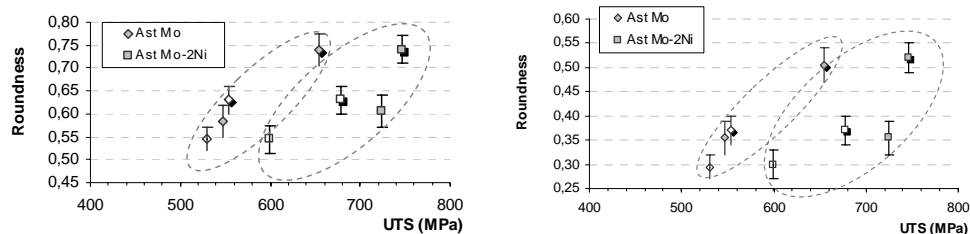

Fig.5. Pore area vs. UTS regarding to processing parameters. Left: taking into account the whole pore system. Right: Taking into account the 10 % of bigger pores.

Also noticeable is the increase of tensile strength for Ni alloyed materials as a consequence of a favourable microstructure, both from the metallographic viewpoint (Ni-rich retained austenite can act as crack barriers) and from the porosity viewpoint, as was shown in Fig.3 (smaller pore areas for Ni-containing materials).

To establish the effect of thermo chemical treatment, materials were sintered under industrial conditions and treated in large scale as well. When materials are carbonitrided, the tensile response is improved and keeps the same relation with roundness. A more favourable pore roundness together with phase and composition changes assist in improving tensile response. HVC is the method that better contributes to this enhancement.
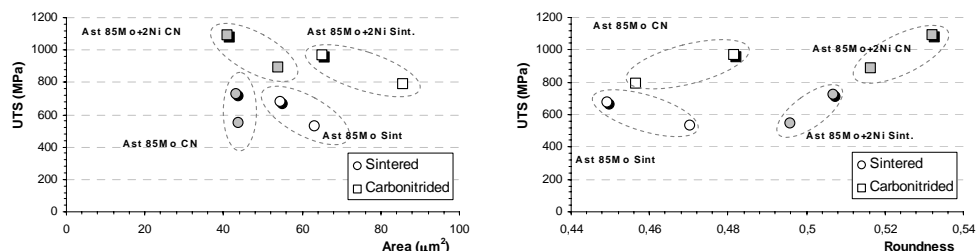

Fig.6. Sintered vs. Carbonitrided properties.

When the comparison is established between as sintered materials (mostly bainitic microstructure) and carbonitrided ones (mostly low-baintic and martensitic microstructure)

(Fig.7), the increase of tensile strength in favour of the latter experiment is mainly due to the microstructural difference, being the pore feature effect not so obvious as to discuss. Solid solution hardening effect of Ni and Mo is also clear in Fig.6, in terms of mechanical properties. An increase in Ni and Mo gives an improvement of about 43 %.
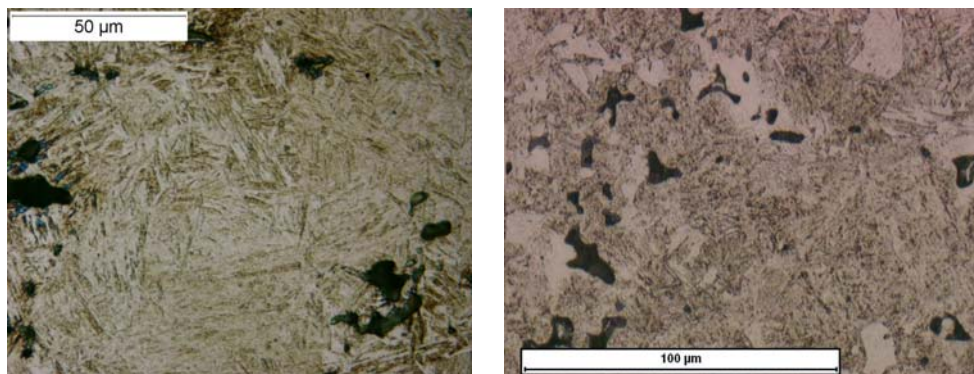

Fig.7. Microstructures of Carbonitrided materials.

CONCLUSIONS

The use of statistical techniques to analyse the measured pores features by image analysis, have been demonstrated to be very useful to allow quantitative comparisons between pore features. Therefore it is possible to establish reliable relationships with the mechanical properties.

It has been shown that a substantial modification in microstructure due to the presence of nickel, will improve the tensile properties since nickel, among other things, acts to reduce the pore area and length. Comparing both densities, it is noticeable that HVC improves the pore system with smaller pores (in average area), so far, with a smaller maximum length and being significantly more rounded, so they are less damaging. It has been stated that this tendency is also maintained when studying just the top 10 % bigger pores, which due to their size can be the most dangerous and relevant in the mechanical properties. It has also been shown that the higher the sintering temperature, the more enhanced the differences in pore features are when comparing both densities.

A study of just the 10 % coarser pores can be enough to predict mechanical properties. Statistical results correlated with the experimental observations, so it is a suitable way for analysing the results.

Thermo chemical treatment, such as carbonitriding, can affect the pore structure; especially in systems where the porosity is mainly open (NC). The diffusion process at the austenitizing temperature is able to close the smallest pores of the system. As a consequence, special care has to be paid to carefully control the case depth.

Acknowledgements

Dedication

Accepting the invitation of the PMP editor to contribute a paper to the issue of the Journal dedicated to Dr. Šalak and his 80th birthday, we have tried to summarize our last two communications [13,14] presented in the last outstanding International Conferences on PM held in Europe, and which deal with the favourite topic of Prof. Šalak: the low alloy PM steels.

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