PHOSPHORUS IN SINTERED STEELS: IMPACT FRACTURE BEHAVIOUR OF SINTERED Fe-P WITH VARYING P CONTENT

V. Vassileva, D. Krecar, C. Tomastik, Ch. Gierl-Mayer, H. Hutter, H. Danninger

Abstract
Specimens Fe-x\%P with varying phosphorus content 0.0 ... 1.0 mass\% were manufactured by mixing plain atomized Fe powder with Fe_3P, uniaxial die compaction to form Charpy impact test bars, and sintering in H_2 at 1120 and 1250°C, respectively. The unnotched bars were impact tested at room temperature, and fractographic studies were performed. It showed that above a critical threshold of the phosphorus content, typical intergranular brittle fracture dominates, resulting in very low impact energy values, while low-P materials fail in a ductile manner with considerable plastic deformation. This effect of the P content is very similar for both sintering temperatures. Investigation of metallographic sections by secondary ion mass spectrometry (SIMS) did not show marked P segregation to the grain boundaries. With Auger electron spectrometry (AES), in contrast, P segregation at the intergranular faces could be clearly identified. Sintering of Fe-P for different lengths of time showed that the intergranular fracture typical for this material becomes more pronounced with longer sintering times, in the early stages of sintering P apparently being distributed inhomogeneously which results in only local clusters of intergranular failure.

INTRODUCTION
In powder metallurgy, the iron – phosphorus system has been studied and described for some time, starting as early as 1940 [1-5], and phosphorus alloyed steels have been investigated thoroughly by numerous research groups (e.g. [6-18]). In contrast to classical ferrous ingot metallurgy, for which P is a highly undesirable impurity, in powder metallurgy phosphorus is intentionally added as an alloying element, in order to exploit the advantages of this system.

All studies indicate that phosphorus accelerates the sintering process in iron, as described e.g. in [14]. The main reasons for this are: 1) during the sintering a liquid phase is formed by a eutectic reaction at relatively low temperatures, as evident from the Fe-P phase diagram [18], and 2) stabilisation of the α-iron, in which the atoms have a high diffusivity, self-diffusion of iron being about two orders of magnitude compared to austenite [8, 10]. Furthermore, this leads to higher sintered densities and to rounding of the pores in the
sintered materials [9], both of which positively influence the mechanical properties [9, 15, 16], increasing the load-bearing cross section [19] and thus improving both strength / hardness and ductility, which is a highly desirable effect compared to e.g. classical alloying or heat treatment which tend to improve strength but at the expense of ductility.

The question also arising in powder metallurgy is where the limits for the positive effects of phosphorus are, in particular at which P content the positive effect reverses to a negative one. The main problem is which role does the phosphorus play in the iron alloy and how is P distributed. There exist different ideas and suggestions about the behavior of the phosphorus [7, 9, 11-13]. It is however agreed in the literature that at P levels >0.6 mass% there is an increased risk of intergranular embrittlement. These effects are attributed e.g. to the transient liquid phase which leaves oxide traces at the grain boundaries [10] or to impurities contained by the ferrophosphorus used [20].

The present study provides a further contribution to the explanation of the behavior and influence of phosphorus in ferrous materials. While the sintering parameters were held constant, the phosphorus concentration was varied over a wide range. Inhomogenities, precipitations or segregations of the phosphorus should be found with the help of selected properties (hardness, impact energy), metallographic and SIMS investigations as well as fracture surface and AES analyses.

**EXPERIMENTAL PROCEDURE**

Iron samples with varying phosphorus content, namely 0; 0.15; 0.30; 0.45; 0.6; 0.8 and 1.0 mass percent, were prepared by the powder metallurgy route. Water atomised iron powder ASC 100.29 (Höganäs AB) and fine (40 µm) ferrophosphorus powder Fe₃P (supplied by MIBA Sinter Austria GmbH) were mixed as starting materials, and 0.5% microwax C (ethylene bisstearamide) was admixed as pressing lubricant. Dry mixing was done for 60 min in a tumbling mixer.

From all the mixes, standard impact test bars 55 x 10 x 10 mm (ISO 5754) were compacted at 600 MPa. The green samples were subsequently sintered in a pusher furnace at 1120 °C / 1250°C, respectively, for 60 minutes under a protecting atmosphere of flowing hydrogen; in a second series, performed with the composition Fe-0.8%P, the isothermal sintering time was varied between 5 and 60 min.

The sintered test bars were broken using a Wolpert Charpy impact tester with \( W_{\text{max}} = 50 \) J at room temperature. The fracture surfaces thus obtained were investigated by SEM in the secondary electron mode. Metallographic samples were prepared by cutting, resin mounting, grinding and polishing with diamond of varying grit for investigations under the light microscope and by secondary ion mass spectrometry (SIMS CAMECA 3f, with Cs⁺ ions for detection of \( ^{31}\text{P}^- \)). The hardness (HV 10) was also measured for these samples. Auger electron spectrometry investigations were done on a Scanning AES VG microlab 310F. Cylindrical samples (d = 5 mm, l = 30 mm) with a circumferential notch were machined from the sintered bars and broken in vacuum. The fracture surfaces thus obtained were investigated by AES; depth profiles were taken by Ar ion sputtering.

**RESULTS AND DISCUSSION**

**Dimensional and mechanical properties**

The results obtained, as presented in Table 1 and 2 which summarise the mean values of several samples, clearly show that the dimensional and mechanical properties depend on the phosphorus content.
Tab.1. Properties of ASC 100.29 - x% P (as Fe₃P), compacted at 600 MPa, sintered at 1120°C, 60 min in H₂

<table>
<thead>
<tr>
<th>P content [mass%]</th>
<th>Green Density [g.cm⁻³]</th>
<th>Sintered Density [g.cm⁻³]</th>
<th>Hardness HV10</th>
<th>Impact Energy [J.cm⁻²]</th>
<th>Dimensional Change [%]</th>
</tr>
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<tr>
<td>0.00</td>
<td>7.05</td>
<td>7.12</td>
<td>54</td>
<td>44</td>
<td>-0.39</td>
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<td>0.15</td>
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</tr>
<tr>
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<td>6.89</td>
<td>7.24</td>
<td>154</td>
<td>2</td>
<td>-1.43</td>
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</tbody>
</table>

Tab.2. Properties of ASC 100.29 - x% P (as Fe₃P), compacted at 600 MPa, sintered at 1250°C, 60 min in H₂

<table>
<thead>
<tr>
<th>P content [mass%]</th>
<th>Green Density [g.cm⁻³]</th>
<th>Sintered Density [g.cm⁻³]</th>
<th>Hardness HV10</th>
<th>Impact Energy [J.cm⁻²]</th>
<th>Dimensional Change [%]</th>
</tr>
</thead>
<tbody>
<tr>
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<tr>
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</tr>
<tr>
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<tr>
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<tr>
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<td>7.40</td>
<td>130</td>
<td>1</td>
<td>-1.41</td>
</tr>
<tr>
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<td>7.38</td>
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<td>-1.39</td>
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<tr>
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<td>7.37</td>
<td>166</td>
<td>&lt;1</td>
<td>-1.41</td>
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</tbody>
</table>

Density

The green and sintered densities of the studied samples are shown in Fig.1 as a function of the phosphorus content, as is the dimensional change.

![Density](image1)

![Dimensional change](image2)

Fig.1. Green and sintered density and dimensional change of Fe-P as a function of the P-content.
As can be clearly seen, the compactibility of the powder mixes decreases with higher P content. The green densities decrease quite markedly with higher amount of P despite constant pressure being applied, as a consequence of the fine and fairly hard ferrophosphorus powder used.

On the other hand, the phosphorus content activates the sintering processes, more P resulting in an increase in the amount of low-melting transient eutectic present and through stabilisation of the ferrite, and leads to an increase in the sintered density. This is evident from the high density of 7.24 g.cm\(^{-3}\) attained with 1\% P, which is also linked to considerable shrinkage, while the phosphorus-free samples only reach the density of 7.12 g.cm\(^{-3}\) in the sintered state and exhibit only slight shrinkage. According to [18], at 1120°C the plain austenite region is left at P > about 0.27 mass\%, and plain ferrite region is attained at – depending on the respective reference – 0.55 to 0.66 mass\% (Raghavan [21] gives a maximum of 0.30 / 0.68 mass\% at 1150°C), which agrees well with the shape of the sintered density graph shown above: upon entering the two-phase region the density tends to increase, and after attaining the single-phase ferrite range there is only slight increase of the density with higher P content.

Here it should also be considered that the addition of P lowers the solidus temperature of the material, resulting in higher relative sintering temperature compared to P-free reference material for the same nominal temperature. From [18] can be derived that the solidus temperature decreases from 1538°C for plain Fe to about 1420°C for Fe-1.0\%P, which means that for 1120°C the relative sintering temperature (\(T_{\text{sint}}/T_{\text{sol}}\), both in Kelvin) increases from 0.77 to 0.82, which latter value for plain iron would correspond to a sintering temperature of about 1210°C. Therefore, also the pronounced effect of P on the solidus temperature of ferrous systems should not be ignored, although from Fig.1 it can be concluded that the presence of ferrite is the main parameter affecting densification during sintering.

**Mechanical properties**

The mechanical properties of sintered iron-phosphorus alloys are illustrated in Fig.2 which shows impact energy and hardness as a function of the phosphorus content. As can be clearly seem, the hardness increases linearly with the phosphorus content and is about twice as high after addition of 0.6\%P as compared to plain iron. The hardness values increase from HV 55 for plain iron to HV 117 with 0,6\% P and reach a maximum of HV 154 at 1\%P.

![Fig.2. Impact energy and hardness as function of P content.](image-url)
This pronounced increase of the hardness is mainly a result of the powerful solid solution hardening effect of phosphorus in the ferrite lattice (and also explains why adding phosphorus by prealloying is avoided in practice).

The impact energy remains at first unchanged with P contents up to 0.15 mass%. Above 0.15 mass% P, the impact energy decreases continuously and quite significantly but the hardness increases only linearly which results in a relationship impact energy – hardness as shown in Fig.3.

![Fig.3. Impact energy of Fe-x%P plotted vs. Hardness.](image)

Although a consolidation and densification of the samples and a rounding of the pores takes place at higher P content, whereby a general increase in the ductility would be expected, in the Fe-P system the impact energy is reduced quite dramatically if the phosphorus content increases from 0.3% to 1%, and this loss of ductility cannot be attributed to the higher hardness of the ferrite, sintered steels with similar hardness as e.g. Fe-0.8%C typically showing impact energy values in the range of 10 ... 20 J.cm\(^{-2}\).

The fact that impact energy and hardness at phosphorus contents above 0.15% no longer change in the same way can be taken as an indicator for the onset of intergranular embrittlement (Fig.5). Phosphorus contents in excess of about 0.3% result in an increased shrinkage rate, as clearly visible from Fig 1b. The shrinkage increases very rapidly with higher phosphorus content due to the high self-diffusivity of iron in ferrite and the presence of liquid phase which is formed above 1050 °C [18]. This results in enhanced phosphorus activation at the grain boundaries, but after cooling might result in embrittlement there, also as a consequence of the segregation tendency of P well known from ingot metallurgy.

**Microstructural and SIMS investigations**

The metallographic investigations show that the pore structure of the phosphorus-free samples is angular and quite irregular (Fig. 4a). An increase in the phosphorus content leads to a rounding and coarsening of the pores through liquid phases formed during sintering and reduces the total porosity (Figs.4b-d).
After Nital etching it can be seen that plain iron materials have significantly smaller ferrite grains (Fig.4e) than the samples with 0.6%P (Fig.4f). This grain growth can be attributed to the ability of the phosphorus to stabilise the α-state of iron in which the diffusivity of the atoms is approximately two orders of magnitude higher than in austenite [10]. No segregations or precipitates of P can be observed at the grain boundaries.

SIMS investigations were performed on unetched (ground and polished) specimens. The phosphorus appears to be homogeneously distributed and there was no detectable evidence of precipitates or segregation at the grain boundaries, which agrees with findings described e.g. in [22]. Of course it must be considered here that very thin segregation layers e.g. at grain boundaries cannot be detected in metallographic sections unless the resolution is in the low nm range, which is not the case with SIMS. For this purpose, analysis of the fracture surfaces is better suited if analyzing techniques with high surface sensitivity such as AES are used.

Fractographic analysis by SEM and AES

The fracture surfaces of the alloys with varying P contents obtained from impact tests were examined by SEM in secondary electron mode and compared to those of plain iron samples.

As expected, plain iron shows a ductile fracture surface, the broken sintering necks being characterized by a fine dimple structure (Fig.5a). For the P containing specimens
quite interesting effects were observed: Although it is described in the literature [1, 10, 12, 13] that small amounts of phosphorus, at least up to 0.45 mass% [23], lead to an increase in both strength and ductility, it is interesting to see in the present fractographs that individual, localized areas of brittle intergranular fracture can be observed already at a phosphorus content as low as 0.3%P (Figs.5b, 5e). Although ductile fracture still dominates, these brittle areas already affect the impact energy adversely, which is clearly evident from Fig.2a which indicates a drop of the impact energy between 0.15 and 0.30 mass%.

If the P content is increased further, the material becomes more and more brittle, and at higher P levels purely intergranular brittle fracture is observed (see the sequence Fig.5a-d). This purely intergranular fracture mode is particularly evident with the specimens sintered at 1250°C, mainly because of the coarser grains and resulting larger facets (compare Figs.5d and 5f). In particular from this appearance of the fracture surfaces the very low impact energy is well comprehensible.

As stated above, neither metallography nor SIMS analysis of sections revealed any precipitates or P-segregation, which indicates that the embrittling layers, if present, should be very thin; it is well known that even monoatomic layers at interfaces can already cause embrittlement. Therefore it was necessary to find other methods which could explain the decrease in the impact energy values and the occurrence of brittle fracture, i.e. which are able to detect even monoatomic layers. Here, AES was regarded as the method of choice [24].

The SIMS-technique has a very high analyzing range, i.e. extreme sensitivity for trace elements. Auger electron spectroscopy, on the other hand, is much less sensitive regarding the concentration but excellent regarding vertical resolution and is suitable even for the analysis of monolayers. Therefore, AES spectra were taken on brittle and ductile areas, respectively, of the fracture surfaces (marked P1 and P2 in Fig.6a).
Fig. 5. Fracture surfaces of Fe-x%P, compacted at 600 MPa, sintered for 60 min at varying temperatures in H₂.
Fig.6. AES analysis on impact fracture surfaces of specimens Fe-0.3%P/Fe-0.8%P, sintered 60 min at 1120°C in H₂.

In Figure 6, Auger spectra of intergranular-brittle and ductile fracture surfaces, respectively, of Fe-0.3%P are shown. The spectrum taken on the intergranular fracture surface (P1 in Fig.6a) clearly demonstrates the enrichment of phosphorus (Fig.6b lower
The graph contains a P-peak, but the other spectrum (Fig.6b, upper graph, from P2 in Fig.6a) does not exhibit a P peak.

Of course, here a fundamental weakness of the fractographic studies by AES should be considered, i.e. that grain boundary segregation can be detected only if failure occurs in fact at the grain boundaries [25]. Therefore it can be proved that P segregates to the failed grain boundaries but it cannot be shown that the grain boundaries in the areas with ductile fracture are free of P segregation, i.e. there is no accessible reference area. Regarding the pronounced differences between the failure modes evident here and also the well known tendency of P to segregate to the grain boundaries and embrittle them, it can however be safely concluded that P segregation is in fact the reason for the intergranular failure observed here.

The corresponding concentration-depth profile of P1 (Fig.6c) shows that P is enriched at the grain boundary, while the corresponding Fe-concentration at the same site is comparatively low (left part of the diagram) but increases with longer sputtering, indicating that the P enriched layer is rather thin.

An Auger spectrum and a concentration-depth profile were also measured for a sample with 0.8%P which exhibited fully intergranular failure (Fig.6d-f). Fig.6e shows that phosphorus has been enriched at the fracture surface, and the corresponding depth profile (Fig.6f) has a broader P peak than in Fig.6c, i.e. the phosphorus concentration in the areas of brittle fracture is higher than in the Fe-0.3%P material, which agrees with the higher total P content.

**Influence of the isothermal sintering time**

Since P was added as admixed ferrophosphorus, its distribution could be expected to be initially heterogeneous. The metallographic and fractographic investigations described above had shown that intergranular failure tends to start in clusters here, indicating chemical heterogeneities and thus heterogeneous P distribution. It was therefore expected that long-range homogenization of P would occur in the course of sintering.

In order to check this hypothesis, sintering runs were done for varying lengths of time, the sintering temperature being kept at 1120°C, and the P content was chosen as 0.8%, since in this case the activating as well as the embrittling effects were expected to stand out particularly clearly. Dimensional properties as well as the impact energy were determined (see Table 3), and metallographic and fractographic studies were done.

**Tab.3. Properties of ASC100.29 - 0.8% P (as Fe₃P), compacted at 600 MPa, sintered at 1120°C in H₂ for different lengths of time**

<table>
<thead>
<tr>
<th>Sintering time [min]</th>
<th>Green density [g.cm⁻³]</th>
<th>Sintered density [g.cm⁻³]</th>
<th>Dim. change [%] linear</th>
<th>Impact energy [J.cm⁻²]</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>6.94</td>
<td>6.99</td>
<td>-0.25</td>
<td>1.79</td>
</tr>
<tr>
<td>10</td>
<td>6.93</td>
<td>7.05</td>
<td>-0.42</td>
<td>1.50</td>
</tr>
<tr>
<td>15</td>
<td>6.92</td>
<td>7.07</td>
<td>-0.42</td>
<td>1.70</td>
</tr>
<tr>
<td>20</td>
<td>6.93</td>
<td>7.07</td>
<td>-0.58</td>
<td>1.77</td>
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<td>60</td>
<td>6.94</td>
<td>7.19</td>
<td>-1.06</td>
<td>2.21</td>
</tr>
</tbody>
</table>
As can be clearly seen from Table 3, there is almost linear increase of the density and, logically, also a similarly linear increase of the shrinkage with longer isothermal sintering. The impact energy remains at very low levels, which is not surprising regarding the fairly high phosphorus content; in any case however this shows that phosphorus embrittlement occurs simultaneously with neck growth since the formation of more massive sintering contacts does not result in higher impact energy values.

The microstructures (Fig.7) show progressive pore rounding; starting from 15 min, densification “clusters” seem to appear, probably as a consequence of local P enrichment, which is also corroborated by the presence of grain boundary phases which with longer sintering tend to appear at all grain boundaries (compare Fig.7b and c).

![Metallographic sections of Fe-0.8%P, sintered at 1120°C in H\textsubscript{2} for different lengths of time.](image)

Fig.7. Metallographic sections of Fe-0.8%P, sintered at 1120°C in H\textsubscript{2} for different lengths of time.

The fracture surfaces (Fig.8) also support the assumption of densification centers caused by locally higher P concentration: while after sintering at 5 and 10 min microductile fracture dominates, with longer sintering time also other fracture morphologies can be seen: after 15 min, intergranular failure emerges; surprisingly, also some cleavage fracture can be seen, which phenomenon disagrees with the low impact energy values, cleavage in sintered steels indicating relatively strong sintering contacts. Apparently these isolated cleavage areas are too small to have a significant effect on the macroscopic behaviour. With further extended sintering, ductile failure as well as cleavage progressively disappear in favour of intergranular failure, the latter still appearing in “nests” or clusters, as clearly visible in Fig.8e (40 min). Finally, after 60 min sintering time, fully intergranular fracture is observed, which is typical for this material as indicated in the previous chapter. Such densification centers have been described also for solid state and liquid phase sintering of other systems, being caused by heterogeneous green density and/or heterogeneous liquid phase formation [26-28]; in the present case the reason is apparently heterogeneous distribution of the phosphorus carrier.

This hypothesis has also been supported by SIMS analysis of larger areas (see Fig.9): here the 31P signal was taken from metallographic sections. The very heterogeneous P distribution after short sintering time is evident, as is the increasing homogeneity, although also at 20 min heterogeneity in the visible P “network” are found, which agrees with the densification centers revealed by fractography.
It can therefore be concluded that at least after sintering for 60 min, phosphorus is sufficiently evenly distributed to segregate to all grain boundaries in more or less the same intensity, thus causing homogeneous intergranular embrittlement. Of course it has to be considered that in the present study specimen preparation, in particular mixing, has been done in laboratory scale, and in industrial batches a more regular initial P distribution should be expected – in particular if the premixed grade PASC 100.45 is employed –, but the pronounced effect of phosphorus distribution during sintering stands out very clearly from the results presented here.

Fig. 8. Impact fracture surfaces of Fe-0.8%P, sintered at 1120°C in H₂ for different lengths of time.
CONCLUSIONS

It could be shown that by addition of P as fine Fe₃P to plain iron powder, materials with fairly high density, with rounded and closed pores and a relatively coarse grained microstructure of Fe were obtained. Higher sintering temperature resulted in slightly higher sintered density and more pronounced shrinkage. The most pronounced density effects were recorded between 0.3 and 0.45 mass% P.

The hardness increases fairly linearly with higher phosphorus content and is doubled by addition of 0.6%P compared to equally prepared plain iron, indicating pronounced solution strengthening. The impact energy of sintered Fe-P, on the other hand, initially increases with addition of phosphorus, reaches a maximum at around 0.15%P and then pronouncedly drops at higher phosphorus contents. Materials sintered at 1250°C tolerate slightly more P than those sintered at 1120°C but the drop to very low values with increasing P contents is more pronounced. Fractographic studies showed a transformation from a ductile, dimple-type fracture of the plain iron to a mixed fracture at 0.3 %P exhibiting some intergranular failure - although the ductile fracture still dominates - to purely intergranular brittle fracture at 0.8 %P, which explains the very low impact energy values.

Auger electron spectrometry studies and the corresponding concentration-depth profiles indicate that even at 0.3 %P, phosphorus is enriched at the grain boundaries – in fact the sintering necks - , at least in the areas of brittle intergranular fracture. At higher P levels, both the area of brittle fracture and the amount of P segregated at the grain boundaries tend to increase.

During sintering of Fe-0.8%P for different lengths of time, at short sintering times localized densification occurs, apparently caused by locally higher P concentrations (as also shown by SIMS investigations), which result in metallographically visible grain boundary phases in these areas as well as in localized intergranular failure, while the remaining fracture surface shows dimple and occasionally cleavage fracture. At longer sintering times, uniform fracture morphology, typically as intergranular failure, is observed, indicating homogeneous phosphorus distribution.

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REFERENCES