The role of phosphorus in pore rounding of sintered steels

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Abstract: The contribution of the surface active element Phosphorus (P) in pore rounding in sintered steels is absent in popular sintering models. Instead, these mathematical models analyze the rate of mass transport, which, according to these models, is driven by chemical potential gradients set up by the differences in the curvature of the free surfaces of the compact of contacting particles. To elucidate the effect that P has on rounding of pores in sintering models, an attempt has been made to incorporate surface curvature in Gibbs’ surface thermodynamics. Using the image analysis method, a comparative study of pore morphology of sintered Iron-Carbon-Phosphorus (Fe-C-P) has been performed to study the effect P has on pore morphology. The application of the shape factor has been used to characterize the morphology of the pores. It was found that morphological enrichment and depletion of P are related to surface stresses as well as to the influence of vacancies.

Subjects: Mechanical Engineering; Manufacturing Engineering; Materials Science; Surface Engineering-Materials Science

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PUBLIC INTEREST STATEMENT

Phosphorous (P), that has two major forms, the white phosphorous and the red phosphorous, never been found as free element because it is very reactive element. Moreover, it is the second most plentiful mineral in your body. In this research, the role that P has on rounding of pores in sintering models has been investigated. An attempt has been made to incorporate surface curvature in Gibbs’ surface thermodynamics. Using the image analysis method, a comparative study of pore morphology of sintered Iron-Carbon-Phosphorus (Fe-C-P) has been performed to study the effect P has on pore morphology. The application the shape factor has been used to characterize morphology of the pores. From this work, it has been found that the morphological enrichment and the depletion of P are related to surface stresses as well as to the influence of vacancies.
1. Introduction

The effect P has on pore morphology of sintered steels has been documented in the experimental literature (Causton, 1996; Danninger, Gierl-Mayer, & Jaliliziyaeian, 2014; Šalak & Selecká, 2012). Unfortunately, this effect is absent from the sintering models (Duva & Crow, 1992) found in the experimental literature. Instead, these mathematical models analyze the rate of mass transport, which, according to these models, is driven by chemical potential gradients set up by the differences in the curvature of the free surfaces of the compact of contacting particles. Generally, sintering models assume that sintering is divided into three stages: initial, intermediate and final stage. In the final stage of sintering, the channel-like pore breaks into isolated pores and surface diffusion is responsible for the phenomenon of pore rounding. Since P is frequently added to PM steels in considerable levels, typically up to 0.6 mass% thus a sintering model that incorporates the rounding effect P has on pores during sintering can be used to tune the effect P has on sintering and optimize mechanical properties of sintered products.

In sintering, surface energy and surface tension are important concepts that determine the morphological evolution of the microstructure including grains, grain boundaries, and pores. In sintered materials thermodynamics dictates that the equilibrium shape of pores, of fixed volume held at isothermal conditions, occurs at minimized total surface free energy, in essence, the process goes in the direction that reduces its total free energy (Bordia & Camacho-Montes, 2012).

The evolution of pores morphology and size are related to vacancy diffusion inside the pores. The presence of different curvatures inside a single pore activates the diffusion of vacancies, thus smoothening of the irregularities in the same pore so rounding of pores is controlled by vacancies diffusion (Pavanati, Maliska, Klein, & Muzart, 2007a; Wan, Shih, & Ilhan, 1995). The vacancies concentration gradient set up by local variation of curvature activates the diffusion of vacancies (DeHoff, 1999). The surface parts, which are convexly curved, are found to have lower vacancy concentration, compared to concavely curved parts of the surface (Barsoum, 2002).

By knowing the two-principle curvatures \( k_1 \) and \( k_2 \) at a given position of the surface, the chemical potential of an atom on the surface will be given by the well-known Laplace equation (Cao, & Wang, 2011):

\[
\Delta \mu = \Omega \sigma (k_1 + k_2)
\]

where \( \sigma \) is the surface energy of the atom position and \( \Omega \) volume per atom in the solid. Equation (1) describes the chemical potential of an atom in a curved surface with respect to a flat plane. Each curvature is taken to be positive if it is convex and negative if it is concave. This means that where the surface is convexly curved the curvature is positive. Thus, higher chemical potential of adsorbent located there and for the concave parts, the chemical potential will be lower than the bulk potential. By using the Gibbs-Thompson-Freundlich equation shown in Equation (2) (Chiang, Birnie, & Kingery, 1997), the equilibrium concentration of vacancies \( c \) for a curved surface based on vacancy concentration for a flat surface \( c_0 \) is:

\[
c = c_0 \exp \left( \frac{\sigma V_m}{RT} \left( \frac{1}{r_1} + \frac{1}{r_2} \right) \right)
\]

where \( r_1 \) and \( r_2 \) are the principle radii of curvature for the curved surface, \( V_m \) is the molar volume, \( \sigma \) is the surface energy, \( R \) is the gas constant, and \( T \) is the absolute temperature. This leads to vacancy diffusion into regions with convex surfaces (vacancy sinks). Consequently, the system reduces its free energy when an atom transfers from the convex to the concave position. This atom diffusion within the pore will lead to smoothening of the inner surface eliminating sharp edges and filling cavities.
Porosity affects mechanical properties of PM parts through geometry and distribution of pores. Roundness of pores is a key geometrical parameter affecting mechanical properties of the sintered PM materials (Makarema, Sabzevara, & Ardakani, 2013). The effect of process parameters temperature, sintering atmosphere, compaction pressure, chemical composition and mechanical properties of the material on the roundness of pore population are considered as the most influential parameters on pore roundness (Abu Jadayil, 2008, 2010, 2011a, 2011b; Abu Jadayil & Alnaber, 2013; Abu Jadayil & Flugrad, 2007; Abu Jadayil & Jaber, 2009; Abu Jadayil & Khraisat, 2010; Abu Jadayil & Mohsen, 2011; Khraisat & Abu Jadayil, 2010; Khraisat, Borgström, Nyborg, & Jadayil, 2009). Higher sintering temperatures leads to rounding of pores due to mainly better bulk and surface diffusion and better reduction of surface oxides. Roundness of pores is also affected by carbon addition since C impose a reductive atmosphere on the compact by means of direct or indirect carbothermic reactions and contribute to oxide reduction of surface particles, improving the Fe–Fe diffusion however C has no direct influence on rounding of pores since it is an interstitial atom which does not affect vacancy diffusion necessary for rounding of pores.

This work will use Gibbs’ surface thermodynamics to explain the effect that P has on the rounding of pores in sintered steels. The considered surface thermodynamics will incorporate the effects of surface curvature.

2. Materials and experiments

The iron base powders used were ASC100 provided by Höganäs AB. Their maximum particle size is 150 μm with an apparent density 3.0 g/cm³. Varying amounts of alloying elements, P, C and Silicon (Si) were mixed with the base powders to yield samples with the compositions shown in Table 1. After preparation, these samples were sintered to study the P effect on the rounding of pores after sintering.

After sintering sample 1 was subjected to post-sintering heat treatment. The levels of total P and total C were 2.44 and 0.32 wt-%, respectively. The heat treatment was carried out for 24 h at 1,000°C the atmosphere was pure N₂ and the samples were cooled in air. This was done to promote solid phase sintering and to dissolve the Fe₃P.

Sample 2 was manufactured by means of single stage die compaction at 500 MPa into common dog-bone type tensile test samples with quadratic cross-section of 6 × 6 mm² and 90 mm. The green density of the die pressed samples is 83%. It was sintered in Nitrogen (N₂) atmosphere for 20 min at 1,250°C followed by decarburization heat treatment was carried out in a tube furnace sealed from both ends having a reducing gas mixture of Nitrogen and Hydrogen (90% N₂–10% H₂). Then, at a temperature of 1,100°C, the moisture content of the atmosphere was increased by letting this gas mixture through a water container at room temperature prior to the furnace while heating continued at the rate of 5°C/min to 1,200°C with a final holding time of 120 min.

Sample 3 contains 0.27% P, 1.6% C and 2% Si to study the effect C has on pore morphology.

Sample 4 was double sintered first at 970°C followed by decarburization then heated up to 1,350°C for 5 min. Decarburization heat treatment was carried out in a tube furnace sealed from both ends, and a reducing gas mixture (90% N₂–10% H₂) was passed through the tube furnace.
The shape factor ($f_{\text{shape}}$) was used for determination of pore morphology:

$$f_{\text{shape}} = \frac{4\pi A}{P^2}$$

where $A$ is the surface area of the pore and $P$ is the perimeter of the pore.

The closer this factor to 1, the smoother and more spherical is the pore (Beiss & Dalgic, 1996). The shape factor was quantitatively measured by image analyzer software. After calibration of the software, random surfaces of each sample were analyzed. The main image analysis steps include: binarization of the image, detection of pore perimeter pixels; discarding of noise and small objects, calculation of pore object perimeter, area, and roundness, creation of roundness and size histograms for relative comparison of imaged samples.

3. Results
The effect $P$ has on the morphology of pores is clearly shown in Figure 1. Here rounded pores are seen whilst pores with a crack like morphology are seen in Figures 2 and 3. The increase in $P$ content and the corresponding displacement of the distribution bell to a higher roundness frequency is consistent with diffusion considerations and can be indirectly related to stronger neck formation. Fracture surface in sample 1 reveal a fascinating set of pore structures. Since almost 100% of the exposed area is intergranular grain surface, these structures provide insights into the inner rounding of pores located at grain boundaries. The roundness of the pore is made up of facets not arcs. The effect of Si on the microstructure can be seen from the microstructures of sample 3 having 2%Si.

Figure 1. Fracture surface of sample 1 after being heat treated showing areas of porosity as well as brittle fracture mode.

Figure 2. Sample 2 sintered first at 1,200°C followed by decarburization.
From Figure 3 it is clear that the graphite forms and it fills the pores without changing the morphology of the pores. Most of the pores are located at grain boundaries having an irregular crack like morphology with no evidence of equiaxed pores.

Figures 4 and 5 show the histograms of the sphericity distribution of the pores in samples 2 and 3 respectively. The value of $f_{\text{shape}}$ is in the range from 0.2 to 0.4. The $f_{\text{shape}}$ shows how smooth the pore contour is, this indicates that in the sintering conditions used for samples 2 and 3, the evolution of pores to a smooth contour is more effective than to a circular form as it was shown in Pavanati, Maliska, Klein, and Muzart (2007b).

These observations are more clearly illustrated by Figures 2 and 3. The results show that the pore morphology shifts towards elongated morphology with increasing C content although the extent of porosity decreases.
Figure 6 shows the microstructure of sample 4 (see Table 1) after decarburization at 1,200°C for 2 h. Figure 7 shows the shape factor sphericity of pores of that sample. Decarburization was done to reveal the morphology of the pores. The pores are mainly concentrated at grain/particle boundaries having an irregular crack like morphology.

4. Discussion

Now we begin by reviewing the thermodynamics of surfaces, without ignoring effects of surface curvature. Any gradient in chemical potential \( \mu \) at solid metal surfaces lead to lateral mass transport, which with time will smear out this gradient (Barth, 2000). The equilibrium condition if several particles are involved is \( \sum \mu = 0 \).

The starting point is the gradient of the Helmotz free energy for a solid surface \( A \). The surface energy \( \sigma \) is related to Helmotz free energy \( F \) by the relation:

\[
F = \sigma A
\]
The surface energy $\sigma$ is related to the partial gradient of $F$ with respect to surface area $A$ by the relation:

$$\sigma = \left( \frac{\partial F}{\partial A} \right)_{T,P,n}$$

where the subscripts denote the quantities being held constant when calculating derivatives. \hfill (5)

The total derivative of $F$ with respect to $A$ is the surface tension $\tau$ which is different from its partial derivative with respect to $A$ that gives surface energy (Ibach, 2006). So the surface tension $\tau$ will be:

$$\frac{dF}{dA} = \tau = \frac{\partial F}{\partial \sigma} \frac{d\sigma}{dA} + A \frac{\partial F}{\partial A} + \sigma = \frac{d\sigma}{d\epsilon} + \sigma$$

This expression is known as total derivative. Mathematically surface tension $\tau$, which is referred to as surface stress in solid surfaces, and surface energy $\sigma$ are different. This means that the solid free surface sustains a state of stress leading to strain energy stretching the free surface without adding new atoms to the surface. The surface stress, which is a second order tensor, is associated with the work against surface deformation, it is the result of forces acting at the material surface. The surface stress can be thought of as an excess of bulk stress. Tensors are quantities which possess both magnitude and direction. The order of the tensor is the number of physical directions associated with the tensor. A second order means that there are two directions associated with the stress, (1) the direction of the force and (2) the direction of the unit normal to the surface which gives the orientation of the surface. Surface stresses are $\perp$ to the surface they act upon thus the surface is in a plane stress condition. The components of the surface stress are shown in Figure 8.
Calculation of the total derivative of $F$ with respect to $A$ does not assume that the other variables are constant instead, it allows the other inputs to depend on $A$.

The Helmholtz excess free energy $F$ and its total differential are defined by Levitin and Loskutov (2009) and given by:

$$F = U - TS$$

(7)

where $U$ is the internal energy and $S$ is the entropy. Differentiating Equation (7) leads to:

$$dF = dU - SdT - TdS$$

(8)

The surface internal energy $U$ has a set of natural variables. $U$ is expressed as $U = U(S, V, N, A)$ and is defined by Dehof (2006):

$$U = TS - PV + \sum \frac{\tau_y}{2} A + \sum \mu_i N_i$$

(9)

The differential quantity $dU$ can be expanded in terms of its partial derivatives according to:

$$dU = TdS + SdT - PdV - VdP + \sum \mu_i dN_i + \sum N_i d\mu_i + A \sum \frac{\tau_y}{2} d\epsilon_{ij} + \sum \frac{A d\tau_{ij}}{2}$$

(10)

Removing the gradient of the independent variables $(T, \mu, \tau)$ we get:

$$0 = SdT + \sum N_i d\mu_i + \sum \frac{A d\tau_{ij}}{2}$$

(11)

And:

$$dU = TdS - PdV + \sum \mu_i dN_i + A \sum \frac{\tau_y}{2} d\epsilon_{ij}$$

(12)

According to the Gibbs–Duhem equation with constant $T$ and $P$:

$$- \sum N_i d\mu_i = \sum \frac{A d\tau_{ij}}{2}$$

(13)

The chemical potential change, connected to a variation of particle number, is due to the surface energy. For a two-component system Fe-I Equation (12) becomes:

$$-\frac{N_j}{A} = - \Gamma_i = \sum \frac{d\tau_{ij}}{2 d\mu_i}$$

(14)

where $\Gamma_i$ is the specific surface excess number of moles of component $i$. This is often referred to as adsorption. Free surfaces tend to minimize the surface Gibbs energy to obtain the most thermodynamically stable surface. Segregated atoms from the bulk to free surfaces are called the adsorbate and the host atom is the adsorbent. $\Gamma_i$ sign +ve or -ve depending on if the species segregates or depletes at the surface. The surface stress governs the macroscopic energetics of segregation phenomena. The reason for the reduction in the surface stress when adsorbates like P adsorb at the pore surface is that the adsorbants replace some of the Fe atoms in the surface and the forces of attraction between adsorbents and Fe atoms are less than those between two Fe atoms, hence the contraction force is reduced. Or it replaces some of the vacancies and the forces of attraction between
adsorbents and Fe atoms are larger than those between Fe and vacancy, hence the contraction force is increased. Rearranging Equation (14) and dividing by $\partial(k_{1} + k_{2})$ Equation (15) is obtained:

$$-\Gamma \frac{\partial \mu_{i}}{\partial(k_{1} + k_{2})} = \sum_{\bar{\nu}} \frac{\partial \tau_{\bar{\nu}}}{2\partial(k_{1} + k_{2})}$$  \hspace{1cm} (15)$$

Surface diffusion is attributed to the various lateral interactions developed on the surface. Elastic strains induced in the surface is a kind of indirect interaction connected within the bulk caused by the adsorbed atoms (Yang, Wang, & Zhao, 2008). Thus the region of the pore surface with high surface strain will have the highest contribution to the chemical potential.

The effect of surface curvature and surface strain on the free surface of a pore can be related to the atomic bonding between the surface atoms (Biener et al., 2009). Considering the convex parts of the surface the atoms has fewer neighbors thus fewer bonds and the surface atoms can stretch out. Using Equation (15) and dividing by $\partial \Gamma$:

$$-\Gamma \frac{\partial \mu_{i}}{\partial \Gamma} = \sum_{\bar{\nu}} \frac{\partial \tau_{\bar{\nu}}}{\partial \Gamma}$$  \hspace{1cm} (16)$$

The chemical potential of the adsorbate is assumed to depend linearly on the curvature of the pore surface according to:

$$\sum_{i} \mu_{i} = \sigma \sum_{i} (V_{m}(k_{1} + k_{2})) + \mu_{wi}$$  \hspace{1cm} (17)$$

where $\mu_{wi}$ is the chemical potential of the adsorbate $i$ for a flat surface and differentiating yields:

$$d\mu_{i} = \sum_{i} V_{m}\sigma d(k_{1} + k_{2}) + \sum_{i} V_{m}(k_{1} + k_{2})d\sigma$$  \hspace{1cm} (18)$$

Using Equations (16) and (18) and dividing by $d(k_{1} + k_{2})$ we get:

$$-\sigma \sum_{i} V_{m} \Gamma_{i} = \sum_{\bar{\nu}} \frac{\partial \tau_{\bar{\nu}}}{\partial(k_{1} + k_{2})}$$  \hspace{1cm} (19)$$

This is the adsorption induced stress change. $\Gamma$ sign +ve or -ve depending on if the species segregates or depletes at the surface. Adsorbates and vacancies as well lead to surface stress change according to Equation (19). For convex regions P segregates to induce a compressive stress as to oppose the tensile stress in the convex region. According to thermodynamics segregated P atoms replaces and deplete existing vacancies at the surface. However, in the concave region species depletes at the surface and vacancies enrich to induce a tensile stress to oppose the compressive stress in the concave region. The total induced stress from segregated atoms and vacancies at the surface will be:

$$-\sigma \left( V_{p} \Gamma_{p} - V_{\text{vacancy}} \Gamma_{\text{vacancy}} \right) = \sum_{\bar{\nu}} \frac{\partial \tau_{\bar{\nu}}}{\partial(k_{1} + k_{2})}$$  \hspace{1cm} (20)$$

From Equation (20) the expression between parentheses \( V_{p} \Gamma_{p} - V_{\text{vacancy}} \Gamma_{\text{vacancy}} \) determines whether or not the segregant will induce a tensile or a compressive stress. Whenever $\Gamma_{p} < \Gamma_{\text{vacancy}}$ a tensile stress will be induced and whenever $\Gamma_{p} > \Gamma_{\text{vacancy}}$ a compressive stress will be induced.

At the concave region the value of $V_{p} \Gamma_{p}$ has to decrease to induce a tensile stress countering the already existing compressive stress in the concave region. So at the concave regions P is depleted. At the convex region the value $V_{p} \Gamma_{p}$ in Equation (20) has to increase to induce a compressive stress.
stress counteracting the already existing tensile stress in the convex region. So at the convex region the vacancies are depleted thus P is enriched.

Another important conclusion from Equation (20) is that the induced stress value depends on the type of the segregated atom whether it is a substitutional or interstitial atom. In the case where the segregated atoms are interstitial the value \( V_\Gamma \) has no effect. Thus interstitial atoms do not contribute to rounding of pores. If the segregated atom i is an interstitial atom, then the coverage \( \Gamma_i \) takes a high value where atomic layers are needed. This can be clearly seen in Figure 3, were the C atoms fills the pores without changing the pores morphology.

The force can be written as the negative gradient of a potential. The gradient of stress with respect to position is a force represented by a vector at every point in space forming a conservative vector field. If the gradient of stress are summed using Equation (20) with respect to the coordinates \( x, y, z \) which leads to:

\[
\nabla \tau \_i = -\sigma \nabla (k_1 + k_2) \sum_i \Gamma_i V_m
\]

The line integral of conservative vector fields is path independent, i.e. the work done in moving along a path on the surface depends only on the start and endpoints of the path. At equilibrium the force is zero this means that the term \( (k_1 + k_2) \) is either zero or constant with position. According to Equation (21) the gradient of stress will be equal to zero only in two cases the first one is if the surface is that of a sphere and the second case is that of a plane or facets. Facets with lower surface energies will be favored and exposed (Polyanin & Manzhurov, 2007; Wu & Overbury, 2015).

5. Conclusions
The use of Gibbs’ surface thermodynamics to explain the effect that P has on the rounding of pores in sintered steels has led to the following findings:

- Rounding of pores during sintering is a direct consequence of surface mass of transport of substitutional segregants and vacancies attributed to the surface stress gradient set up by the surface curvature of the inner pore surface.

- Phosphorus segregates to convex regions to induce a compressive stress as to oppose the tensile stress in the convex region. Conversely, in the concave region, P depletes at the surface to induce a tensile stress to oppose the compressive stress in the concave region. This will smear out the irregularity of the pore surfaces leading to rounding.

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