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The Role of Microstructure in Mechanical Behaviors of Low-Alloy Sintered Steels

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In this study, microstructural aspects that control fracture and deformation resistance of P/M materials are evaluated. Several low-alloy steels were produced under both experimental and commercial conditions for achieving different matrix phases and porosity levels with varying shape factors. A "porosity map" was constructed and used for a quantified study of the dominant mechanism controlling mechanical behaviors. The role of inclusion gathering and secondary pores, as well as sintering mechanism and alloying method is considered and discussed. The mechanism assessment was performed by using microstructure examination through optical and scanning electron microscopy. The results demonstrate that if the porosity level is lowered up to 7 percent, the matrix structure has a vital effect under high sintering temperature conditions. On the other hand, steels that have higher amounts of porosity ($> 12\%$), especially those sintered at low temperatures, are fractured under pore-control mechanism. In the intermediate range, mix-control mechanism (pore and structure) related to singular defects and production condition was realized. Nevertheless, microstructure inhomogeneity and alloying method as well as chemical composition could have a pivotal effect on the fracture and deformation behavior.

INTRODUCTION

The fracture behavior and deformation response of sintered steel are different compared to wrought materials. The inherent porosity decreases the effective load-bearing cross-section [1,2] and therefore, is considered as a microstructural parameter that controls the mechanical properties. The presence of pores is accompanied by decrease in strength and ductility, crack deflection from the main crack growth direction [3,4], microcracking [5] and crack branching [6]. However, the matrix structure (ferrite, martensite,...) could also have a pivotal effect on fracture resistance especially at a low porosity level.

This dependence of mechanical properties can lead to important practical consequences. For instance, Danninger [7] has shown that pore morphology, in particular pore connectivity, has a pronounced effect on deformation response of P/M steel-high microplasticity and localized deformation was observed during monotonic tensile testing at sintered contacts. In such a case, macroscopic deformation is small. On the other hand, if isolated pores are formed, better mechanical properties and macroscopic ductility are resulted.

Moreover, the effect of singular defects such as slag inclusion or large pores on cyclic properties has been studied [8-10]. It has been shown that according to the defect size, the strength of matrix material or large defects could determine the fatigue limit. Thus, mechanical properties are controlled by pore-matrix interaction related to density, alloying method, purity of starting materials as well as handling, mixing, compacting and sintering of the powders.

Although the relationship between porosity and mechanical properties has been investigated extensively and several empirical equations have been derived [e.g., 11-14], there are still some shortcomings about the co-effects of pores and matrix materials related to production conditions that must be elucidated through further research. In this study, the effects of manufacturing parameters on the mechanical properties of some common sintered steels are investigated. A "pore map" is constructed and used for realizing the microstructural features that control fracture resistance and limit the performance of sintered steels. Moreover,

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some practical remedies for producing high strength materials under commercial conditions are identified.

MATERIALS PREPARATION AND EXPERIMENTAL PROCEDURES

The chemical composition of materials used for this investigation is shown in Table 1. Atomized iron powder (Hogonas ASC 100, 29) was utilized as a homogeneous model material (grade A). Sponge iron powder (Hogonas SC 100, 26) was also used for producing grades B....G steels that performed through mixing elemental powders. Moreover, the effect of alloying method is studied by using atomized Mo-alloyed iron powders (grades H....J).

The powders were blended for 1 hour in a tumbling mixer and then compacted in a pressing machine at a capacity of 300 tone with floating die to form rectangular specimens 90 × 10 × 10 mm^3. High density was obtained through double pressing and double sintering technique. Intermediate annealing was performed in a tube furnace equipped with a microprocessor under flowing H_2-Ar gas for 30 mins at 700°C. The tube was evacuated by a rotary pump before the furnace was heated. The applied compacting pressure for preparing the specimens were more in a range of 450-600 MPa.

Final sintering was carried out in the tube furnace at 1120°C for 60 mins. Also, some of the steels were sintered at 1240 or 1280°C in a high vacuum resistance furnace (10^{-4} torr) for 60 mins after dewaxing in the tube furnace. The mean cooling rate in the phase transformation range was measured by a Pt-Pt/Rh thermocouple attached to the samples. It was 12 and 27 K/min for the tube and vacuum furnace, respectively.

The density of the sintered samples was measured by water displacement method according to recommendation of ISO Standard 2738/1987. Some of the specimens were heat treated by austenitizing at 800-850°C for 30 mins under controlled atmosphere and then quenched in oil and tempered at 300°C for 60 mins in air.

Table 1. Chemical analysis of the materials examined, wt.% (balance Fe).

Material	C	Mo	Ni	Mn	Cr	Cu
A	0.01	-	-	-	-	-
B	0.5	1	2	-	-	-
C	0.6	0.5	2	-	-	4
D	0.6	0.5	4	-	-	2
E	0.6	1.5	-	3	-	-
F	0.6	0.2	-	0.7	1	-
G	0.7	1.5	-	-	-	-
H	0.7	1.5	-	-	-	-
I	0.7	1.5	-	-	-	2
J	2	1.5	-	-	-	-

Laboratory tensile tests were performed on an electro-mechanical testing machine (Instron 1115) at 0.01 s^{-1} strain rate on the samples with 30 mm gage length and 6 mm diameter according to recommendation of ISO Standard No. 2740/1986. Metallography and hardness tests were also carried out on the broken specimens. The difference between ratio of tensile strength to hardness for the samples, here and in the previous investigation [11], is related to different sample geometries used. The tensile test specimens were prepared through ordinary machining and then in accordance with the standard, the surface area of the middle of specimens was finely polished by felt.

Moreover, the fracture surfaces of broken samples under impact testing were examined by scanning electron microscopy. A semi-automatic quantitative fractography technique was used for measuring the effective load-bearing cross-section of broken samples at 77°K. Also, wet chemical analysis was utilized to assess the material loss during processing. It was up to 0.35 weight percent for carbon related to the type of iron powder (sponge or atomized) and the sintering condition. The carbon loss can be due to sintering in an atmosphere that was a mixture of hydrogen and argon in the laboratory. The carbon loss was rather homogeneous.

RESULTS AND DISCUSSION

The summarized results of the study are shown in Tables 2 and 3. The properties of all tested specimens are affected by chemical composition, matrix phase, total porosity, sintering temperature and heat treatment. The effects of manufacturing parameters on fracture strength are explained by a theoretical basis for realizing the microstructural features limiting the performance of P/M materials.

Density Effect

The effect of manufacturing conditions on mechanical properties of the investigated materials is shown in Figure 1. It is obvious that the most important microstructural feature that could have a pronounced effect on the strength of P/M material is porosity level, which is in agreement with other previous work [e.g., 15]. However, the matrix phase also has a drastic effect on the mechanical properties, depending on the density level. After heat treatment, in low density steels ($P > 12\%$), significant changes in the tensile and impact energy are not observed. However, at higher densities, difference between sintered and heat treated materials is relevant (e.g., Figure 1a). The dashed lines

Table 2. Mechanical properties of sintered steel samples.

Steel	Density (g/cm^3)	Sintering Temp. (°C)	UTS (MPa)	HB (kg/mm^2)	Unnotched Impact Energy (J)
B	7.02	1280	476	135	28
B	7.27	1280	500	146	51
C	6.72	1120	585	249	7
D	7.14	1120	1100	204	30
E	6.85	1280	*	160	21
E	7.15	1280	482	179	31
F	6.61	1120	185	84	*
F	6.86	1280	488	147	20
F	7.12	1240	342	-	43
G	6.96	1120	413	155	8
G	7.1	1240	556	-	11
G	7.19	1280	615	171	28
G	7.45	1240	678	185	58
H	6.92	1240	553	150	15
H	7.17	1240	556	-	*
I	6.99	1120	513	207	22
J	6.75	1120	*	210	10

* not reliable data was obtained because of brittleness of the specimens.

Table 3. Mechanical properties of *P/M* steel samples after heat treatment.

Steel	Density (g/cm^3)	Sintering Temp. (°C)	UTS (MPa)	HB (kg/mm^2)	Unnotched Impact Energy (J)
B	7.02	1280	784	249	16
B	7.27	1280	895	306	35
C	6.72	1120	671	255	6
D	7.14	1120	1024	292	16
E	6.85	1280	748	278	6
E	7.15	1280	766	306	17
F	6.61	1120	214	-	*
F	6.86	1280	919	-	7
F	7.12	1240	555	207	14
G	6.96	1120	1065	-	8
G	7.1	1240	1079	326	12
G	7.19	1280	1353	352	23
G	7.45	1240	1438	415	28
H	6.92	1240	718	260	10
H	7.17	1240	1015	-	*
I	6.99	1120	559	-	15
J	6.75	1120	555	415	2

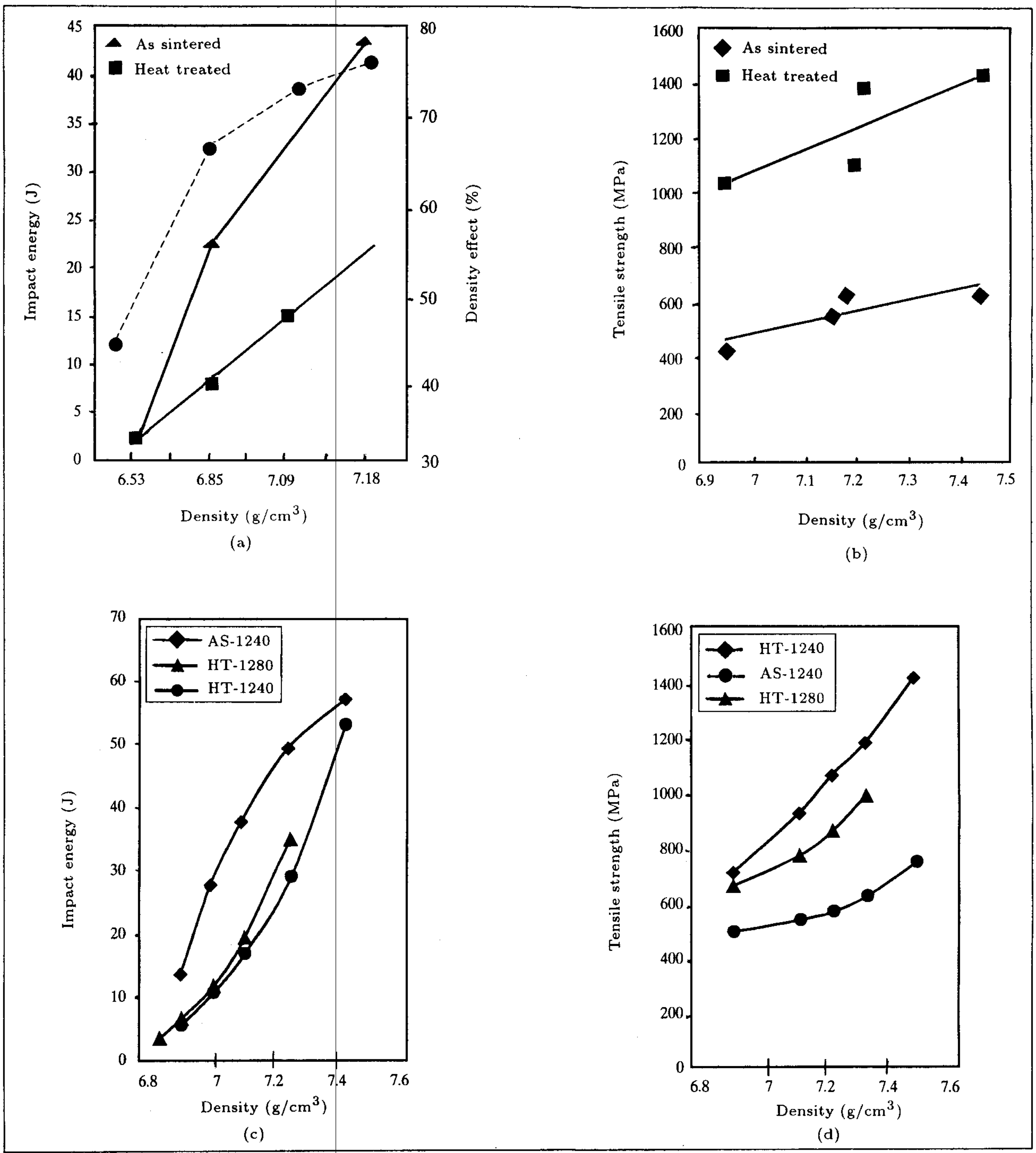

Figure 1. Density dependence of the mechanical properties of some specimens, (a) material F, (b) material H, (c) and (d) various grades (more data can be observed in Tables 2 and 3).

show the density effect (D_e) according to the following definition:

$$D_e = \left| \frac{M_S - M_H}{M_S} \right| \times 100 , \tag{1}$$

M_S: mechanical properties of the sintered sample,
M_H: mechanical properties of the heat treated sample.

This effect is a function of porosity and can be presented by the equation:

$$D_e = f(1 - P) , \tag{2}$$

where P is total porosity.

It is evident that this relationship is not always linear especially for steels which are sintered at lower temperatures (Figures 1c and 1d). Sudden changes in the slope of the curve in medium density range suggest

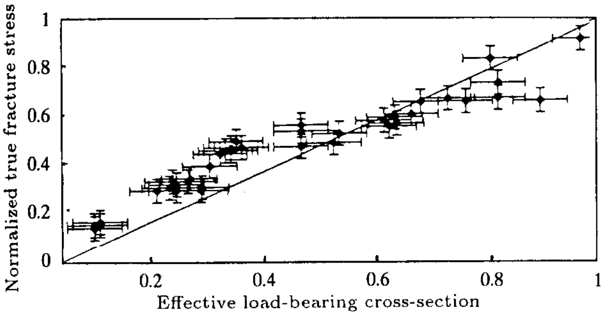

Figure 2. True fracture strength of sintered iron as a function of effective load-bearing cross-section.

that the strength of the materials must be controlled at least by two mechanisms. For low densities, the level of pore characteristics and for high densities, the matrix phase properties have more influence. This dependency for homogeneous matrix phase systems can be interpreted using plain porosity concept stated by Slesar [2]. Figure 2 shows the results for plain iron. However, for inhomogeneous materials, a new microstructural model is required.

Sintering Temperature and Alloying Method

The effect of high sintering temperature on the tensile strength of sintered Cr-alloyed steels is shown in Figure 3. After sintering at high temperatures, a sudden and unexpected change appears; higher density does not mean better mechanical properties. Another reason for decreasing tensile strength could be decarburization at higher temperatures in these experiments.

The influence of sintering temperature on the mechanical properties of *P/M* materials has been studied for several years [e.g., 16]. Pore roundness and increase in material homogeneity through enhanced diffusion activation energy or formation of liquid phase are the most consequential attributes of high temperature sintering. Sintering of Cr-containing steels made from elemental powders at temperatures as high as 1280°C could have a pronounced effect on the mechanical properties because of liquid phase formation [17]. On the other hand, in pre-alloyed starting powders, pore roundness is the creative outcome.

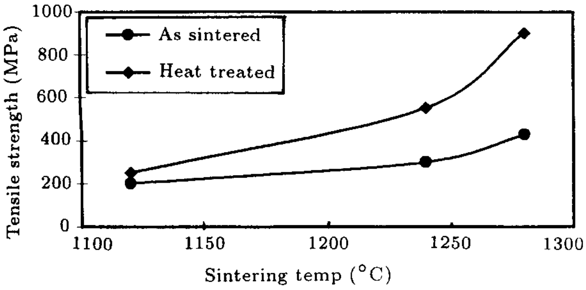

Figure 3. Dependency of the tensile strength with sintering temperatures in sintered and heat treated samples of Cr-containing steels (grade F).

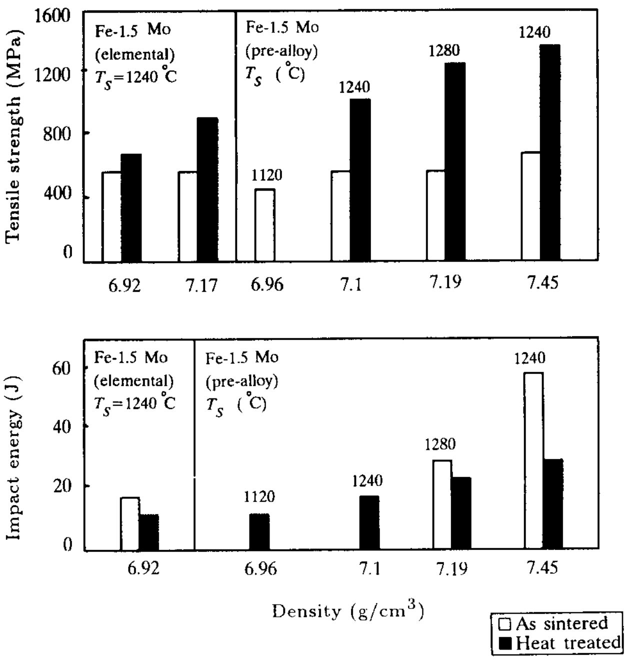

Figure 4. Density dependence of the tensile strength and impact energy of Fe-1.5, Mo-0.7% C steel at various sintering temperatures.

Figure 4 illustrates the effect of the alloying method on the mechanical properties of Mo-alloyed steels produced from elemental and pre-alloyed powders. It is well-known that in the case of elemental starting powders, transient liquid phase forms between Fe-C-Mo in the following sequences [18-20]:

a) Carburization of Mo particle to form Mo_2C,

b) Fe diffusion into the carbide and formation of M_6C (approx. Fe_3Mo_3C),

c) Liquid phase generation between austenite and M_6C.

Therefore, pore roundness and material homogeneity have been achieved through liquid phase formation when sintering is carried out at 1240°C. Under this condition, similar properties for both alloying methods are expected. However, it can be stated that tensile strength is predominantly a function of density, while with impact energy not only density but also the sintering intensity and pore shape play a major role. Sintering mechanism and alloying method are considered to have a pivotal effect on the fracture resistance, however, density should also be taken into account for determination of deformation mechanisms.

Alloying Elements

The effect of alloying elements on the mechanical properties of the investigated steels is illustrated in Figure 5. Toughness is improved through alloying with nickel (note grades B and D), which is in agreement with previous work [e.g., 21], and in high concentrations

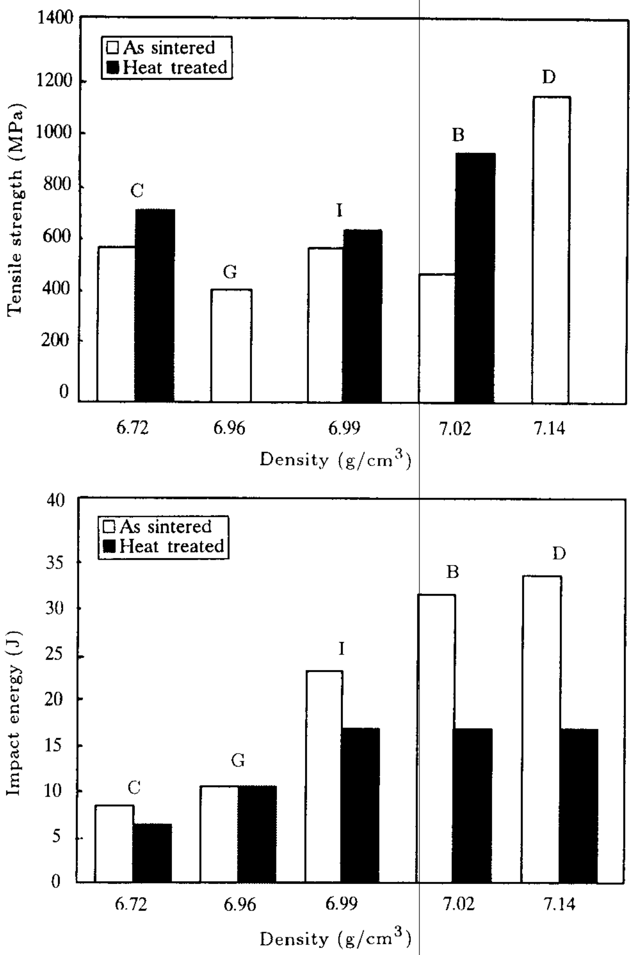

Figure 5. Tensile strength and unnotched impact energy of various investigated *P/M* steels.

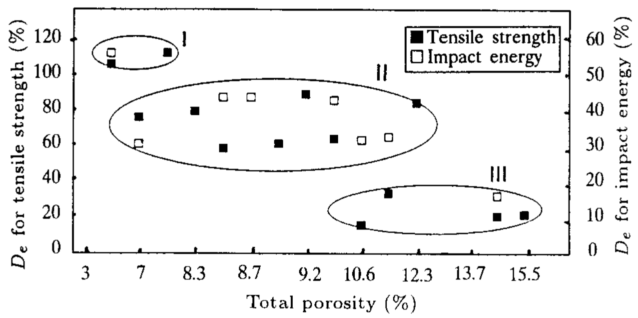

Figure 6. Effect of heat treatment on mechanical properties of the investigated steel as a function of total porosity.

(~ 4%) hardenability of steel rose to a level that hard phases formed during cooling in the sintering furnace. Nickel rich martensite areas and bainitic matrix are formed. The impact energy of the steel decreases after heat treatment unlike hardness. Microstructural examination shows that the bainitic phase is replaced by tempered martensite and due to better impact properties of the bainitic structure [22], hardness increases and toughness decreases. It must be noted that density of the steel is in the range at which matrix structures have a marked influence.

Moreover, during sintering of copper alloyed steels prepared from elemental powders, the combination of transient liquid phase formation and solid solution hardening results in better tensile strength even at lower densities (note grades B and C). Conversely, the impact energy is lower because of sintering in the presence of liquid phase and formation of large secondary pores. Nevertheless, after heat treatment, steels with higher densities (grade B) have superior mechanical properties. Also, copper addition in Mo-steels (grade I) results in better mechanical properties. Nevertheless, a variety of alloying elements and their effects on the sintering mechanism and hardenability of steel as well as its microstructure must be evaluated in addition to porosity level. It is generaly observed that the UTS level for some of the samples is rather low, especially in sintered specimens with low-alloying elements, while for example material D which is more alloyed represents a higher UTS.

Porosity Map

The dependency of mechanical properties on heat treatment has been known for several years: increase in tensile and yield strength and decrease in toughness. However, in *P/M* steels, the severity of these effects is related to density as shown in Figure 6. According to production conditions, three areas are discernible as follows (see Table 4):

I) Small changes in mechanical properties in steels with high porosity ($P > 12\%$) that were sintered at low temperatures ($T_S = 1120°\text{C}$),

Table 4. Effect of heat treatment on the mechanical properties of sintered steels dependent on manufacturing conditions.

Section	Total Porosity [%]	Alloying Method	Sintering Temp. (°C)	Effect of Heat Treatment	Influenced by
I	> 12	Various types	Low ($\geq$ 1120)	Low	Pore
II	7-12	Elemental	High ($\geq$ 1240)	Medium	Metal and pore
III	< 7	Pre-alloyed	High ($\geq$ 1240)	High	Matrix phase

II) Intermediate case in steels made from elemental powders sintered at high temperatures with medium density,

III) Large improvement in tensile and impact strengths in pre-alloyed powders with high density ($P < 7\%$) sintered at high temperatures ($T > 1240°C$).

In *P/M* steels, pores are not affected by hardening treatment and, therefore, the load-bearing cross-section determines the fracture behavior. However, if sintered necks between particles are increased, the influence of heat treatment increases. The dependency of the mechanical properties on these areas can be understood more clearly from Figure 2. Low sintering temperatures cause interconnected pores and isolated sintered contacts that are fractured accompanied by large microplasticity (Figure 7). Under this condition, hardening of the steels could not produce high performance materials because of the low load-bearing capacity of small sintered necks. In contrast, in steels with high densities or those sintered at intense conditions (high temperature or long time), isolated pores are found (Figure 8) and, thus, the mechanical properties are further affected.

However, there are some steels in the intermediate region with low porosity levels ($P \sim 7\%$) that were sintered at high temperatures. In an optical and electron scanning study of the fracture surface, inclusion gathering and secondary pores are observed (Figure 9). The starting powders were sponge iron with relatively high inclusion content. Also, particles of stable oxides such as manganese oxides are reduced to some degree and form a spongy structure [7]. On the other hand, large secondary pores are formed during liquid phase sintering (Figure 10) that have harmful effects on fracture resistance and deformation response. It must be mentioned that these defects have more influence on dynamic rather than static properties [10].

Figure 7. Fracture surfaces of Fe-1.5 Mo-0.7% C steel sintered 60 mins at 1120°C, density is 6.8 g/cm^3, illustration of large microplasticity at sintered necks.

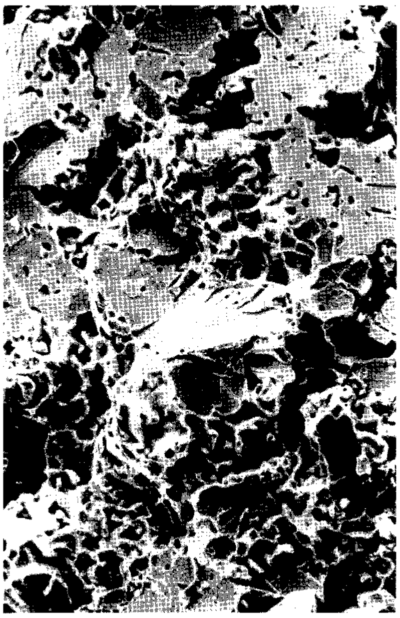

Figure 8. Fracture surface of Fe-1.5 Mo-0.7% C steel sintered 60 mins at 1240°C, density is 7.1 g/cm^3.

From the results mentioned above, a porosity map can be constructed that demonstrates the effects of all important parameters which control the mechanical behaviors. Such a diagram is shown in Figure 11. From this map, it is possible to recommend

Figure 9. Inclusion gathering in a Mn-alloyed steel sintered 60 mins at 1240°C.

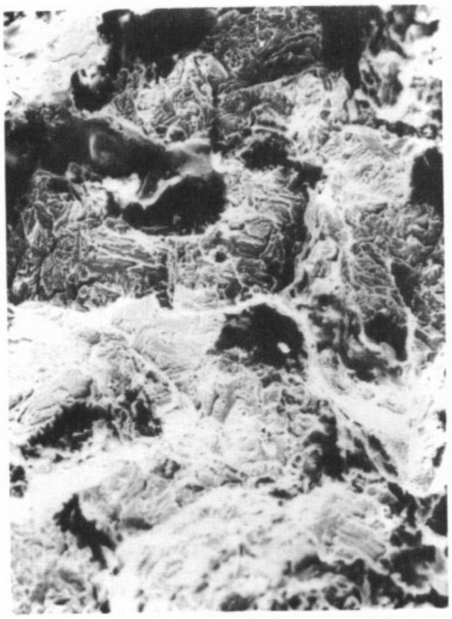

Figure 10. Large secondary pores formed through liquid phase sintering in Fe-1.5 Mo-2% C steel.

the optimum condition for producing high strength steels under commercial conditions. This can be obtained through increasing the density attainable by double pressing or warm compacting route that cause high load-bearing cross-sections. Also, adjusting the chemical composition of steel for sintering under transient liquid phase or intense conditions (high temperature or long time) as well as using powder types with less inclusions is highly recommended.

CONCLUSIONS

1. The severity of porosity effect on the mechanical properties of P/M steel depends on the density level. In high density materials, less dependence of the properties to porosity is found in comparison to lower density materials.
2. The plain porosity can be used successfully for prediction of the mechanical properties of homogeneous matrix phase systems. However, in multicomponent systems, several factors such as alloying method, purity of starting materials as well as handling, mixing, compacting and sintering conditions must be regarded together with load-bearing cross-section for the modeling of mechanical behaviors, especially, dynamic properties.
3. The role of microstructure in the fracture resistance and deformation response of P/M steels can be evaluated by using a porosity map. This map shows the effect of production conditions on the mechanical properties. According to this map, the following three mechanisms control the properties:
   a) If low sintering temperature is used for materials with relatively low densities ($P > 12\%$), interconnected pores/isolated sintered contacts are formed. Under this condition, deformation is concentrated at the contacts and low macroplastic deformation is found.

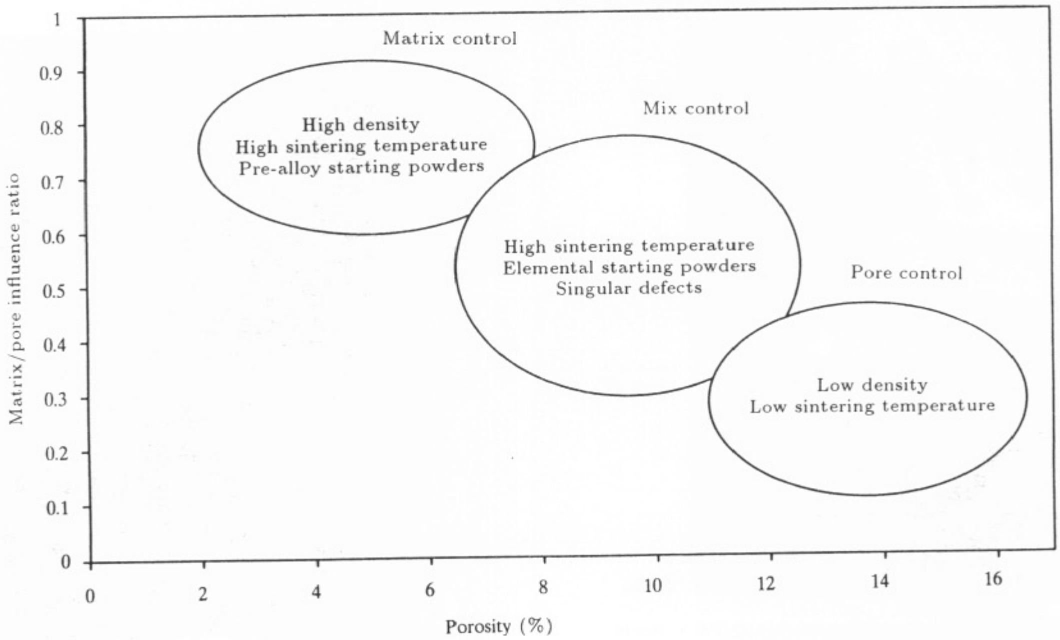

Figure 11. Porosity map for realizing the controlling mechanism of fracture resistance and deformation response of P/M steels.

b) Compacting to a high density ($P < 5\%$) and intense sintering condition result in the formation of isolated pores and high load-bearing cross-section materials. Optimum mechanical properties can be achieved under this condition.

c) Singular defects such as large secondary pores, slag inclusion or unreduced oxides are found in P/M steels as a consequence of liquid phase sintering, insufficiently clean starting materials or improper sintering conditions. These singularities adversely affect the mechanical behavior, especially, dynamic properties. Thus, producing high performance materials is not possible even with high density steels that are sintered under intense conditions.

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REFERENCES

1. Danninger, H. "The effect of microstructure on mechanical properties of P/M steels", *Pract. Metallography*, **31**, p 56 (1994).
2. Slesar, M., Dudrova, E. and Rudnayova, E. "Plain porosity as a microstructural characteristics of sintered materials", *Powder Metall. Int.*, **24**, p 236 (1992).
3. Lindstedt, U., Karlsson, B. and Masini, R. "Influence of porosity on deformation and fatigue behavior of P/M austenitic stainless steel", *Powder Metall. Int.*, **33**, p 57 (1997).
4. Karlsson, B., Wasen, J. and Bertilsson, J. "Fractographic aspects on the fatigue crack growth in porous sintered steels", *Proc. of the 8th European Conference on the Fracture*, **1**, EMAS, Warley, p 421 (1990).
5. German, R.M. and Queeney, R.A., *Metals Handbook, ASM*, **19**, p 430 (1993).
6. Mai, Y., Cotterell, B., He, S. and Ke, Y. "Fatigue crack growth and crack shielding in a Fe-C-Cu sintered steel", *Handbook of Fatigue Crack Propagation in Metallic Structures*, Elsevier Publ., **1**, p 221 (1994).
7. Danninger, H., Spoljaric, D. and Weiss, B. "Microstructural features limiting the performance of P/M steels", *Powder Metall. Int.*, **33**, p 47 (1997).
8. Hadrboletz, A. and Weiss, B. "Fatigue behavior of iron based sintered materials", *Int. Materials Reviews*, **42**, p 45 (1997).
9. Spoljaric, D., Danninger, H., Weiss, B. and Stickler, R. "Fatigue behavior of Mo alloyed sintered steels and its consequences for application", *Proc. of PM Auto' 96*, Isfahan, Iran, p 180 (1996).
10. Spoljaric, D., Danninger, H., Weiss, B. and Stickler, R. "Influence of production parameters on the fatigue properties of low alloyed PM steels", *Proc. PM' 94*, Paris, France, **II**, p 823 (1994).
11. Bocchini, G.F. "The influence of porosity on the characteristics of sintered materials", *Int. J. Powder Metallurgy*, **22**, p 185 (1986).
12. Holmes, J. and Queeney, R.A. "Fatigue crack initiation in a porous steel", *Powder Metallurgy*, **28**, p 231 (1985).
13. Fleck, N.A. and Smith, R.A. "Effect of density on tensile strength, fracture toughness and fatigue crack propagation behavior of sintered steel", *Powder Metallurgy*, **24**, p 121 (1981).
14. Machmeier, P.M., Smith, D.W. and Hendruckson, A.A. "Optimization of toughness in impact forged 46xx steels", *Int. J. Powder Metallurgy*, **29**, p 379 (1993).
15. Danninger, H., Jang, G., Weiss, B. and Stickler, R. "Microstructure and mechanical properties of sintered iron, Part II", *Powder Metall. Int.*, **25**, p 219 (1993).
16. German, R.M. and D' Angelo, K.A. "Enhanced sintering treatments for ferrous powders", *Int. Metals Reviews*, **29**, p 249 (1984).
17. Danninger, H. "Sintering of low alloy steels prepared from elemental powders", *Powder Metall. Sci & Technology*, **4**, p 22 (1993).
18. Danninger, H. and Kara, T. "Influence of manufacturing parameters on the sintering of alloyed structural steels", *Powder Metall. Int.*, **20**, p 9 (1988).
19. Danninger, H. "Secondary porosity in sintered steels and its effects on product quality and consistency", *Powder Metallurgy*, **30**, p 103 (1987).
20. Danninger, H. "Sintering of Mo alloyed P/M structural steels", *Powder Metall. Int.*, **20**(4), p 7 (1988).
21. Bocchini, G.F. "PM steels for advanced application, outlines of selection and design criteria", *Proc. of PM Auto' 99*, Isfahan, Iran, p 43 (1999).
22. Salak, A. "Selecting materials for PM products", *Proc. of PM Auto' 96*, Isfahan, Iran, p 45 (1996).