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Microstructure and Mechanical Reliability of Powder Metallurgy (P/M) Ferrous Alloys

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Because of their macroscopic brittle behavior, porous powder metallurgy (P/M) ferrous alloys are often not considered for structural applications. A statistical approach based on the evaluation and interpretation of the Weibull modulus was thus proposed to evaluate correctly and objectively the intrinsic structural reliability of these materials. In spite of their porosity, P/M ferrous alloys are as reliable as conventional wrought steels, provided that they are correctly produced and, if necessary, heat treated. In addition, the influence of density and the application of the method to the process optimization and control was highlighted. In all cases, the mechanical reliability of the materials was interpreted metallurgically.

Keywords

iron powders, powder metallurgy, reliability, Weibull analysis

1. Introduction

DEPENDING on their deformation and fracture behavior, engineering materials are classified as ductile, semiductile, or brittle. This has a noticeable consequence in designing, especially in the selection of a suitable safety factor. The selected safety factor is usually lower for ductile materials than for brittle ones (Ref 1). Material ductility is often judged by elongation at fracture, and empirical relations link the elongation at fracture to a suitable safety factor (Ref 2).

From a design viewpoint, the safety factor determination encompasses possible higher stresses than the nominal stresses and possible lower material strength than the believed strength. Hence, it has a sound statistical nature and depends on both the characteristics of the loading system and the material (Ref 3). The microstructure and production routes are important material characteristics. For example, the noticeable strength variability of brittle ceramics, which often makes them unreliable for structural applications, is connected to their microstructural flaws. The characteristics of these flaws strongly depend on the processing routes (Ref 4).

In general, material strength distribution is well represented by the Weibull distribution (Ref 5). In its two-parametric form, it is:

$$P(s) = 1 - \exp\left[\int_V -\left(\frac{s}{s_0}\right)^m dV\right]$$

where $P(s)$ is the probability of failure, s is the strength, s_0 is a scale constant, V is the material volume, and m is the Weibull modulus, a material property representative of the distribution shape. In a simplified approach, consider a uniaxial state of stress and, with reference to the maximum applied load, the reliability of a mechanical part, $R(s)$, that is, its probability of survival if submitted to a stress s, is given by (Ref 6):

$$R(s) = 1 - P(s) = \exp\left[-\left(\frac{s}{s_0}\right)^m\right]$$

These bases provide a statistical expression for the safety factor n (Ref 6, 7):

$$n = \frac{\Gamma(1 - 1/m)}{(\ln 1/R)^{1/m}}$$

where Γ is the gamma function. This equation shows that, for a required reliability, R (which essentially depends on the type of application), the safety factor depends on the Weibull modulus only. Moreover, if the m-value is increased, the safety factor decreases. In particular, the safety factor approaches 1 when m is greater than 20 (Ref 6, 7). The value of 20 for the Weibull modulus is often taken as a lower limit for a material in order to have a sufficient intrinsic mechanical reliability (Ref 4, 8). In comparison, soda lime glass has a typical Weibull modulus of 6 (Ref 4). Sintered alumina has a modulus of 12 (Ref 4). Grey cast iron has a modulus of 18. Nodular cast iron has a modulus of 25 (Ref 9). Ductile metals typically have m-values between 30 and 100 (Ref 10).

Powder metallurgy produces low-cost, high-precision components with a wide range of mechanical properties depending on their density and microstructure. Therefore, P/M is suitable for producing mechanical components based on carbon or alloyed steels. However, because of porosity, these materials have a macroscopic deformation and a fracture behavior that is between semiductile and brittle (Ref 11). In particular, they are characterized by very low values of elongation at fracture (even lower than 1% in heat treated alloys, Ref 12) and of impact energy. Consequently, they are often disregarded when structural applications are chosen.

Nevertheless, the micromechanisms of fracture of P/M porous steels are typically ductile, and these materials have collaboration factors higher than unity (Ref 13). Previous investigations (Ref 14-19) proposed evaluating the intrinsic mechanical reliability of these materials by Weibull statistics; i.e., by evaluating

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their Weibull moduli so that the evaluation is direct and objective. This paper reviews some of those investigation results and obtains others in order to demonstrate the metallurgical meaning of the Weibull modulus and the suitability of the approach in P/M process optimization and control.

2. Evaluation of the Weibull Modulus for P/M Alloys

The Weibull modulus for each material was determined with respect to the transverse rupture strength (TRS) distributions relevant to specimens ruptured by three-point bending tests (ISO 3995).

The specimens were 12 mm wide and 6 mm high, whereas the distance between the support points was 25 mm. The TRS was determined with the standard Navier equation. This type of testing was chosen because its simplicity minimizes experimental variations and, in addition, because it allows direct comparison with the literature data.

To ensure a significant statistical analysis, 40 specimens were used for each material type (Ref 20). Tabulated exact median ranks were used to compute the probability of failure, P (Ref 21). The Weibull modulus, m, was then determined by plotting $\ln\{\ln[1/1 - P)]\}$ against lnTRS: the slope of the curve m was determined by the least square method.

Typical of industrial mass production, many materials were included in the experiment. Materials had different chemical compositions and microstructures: ferrite (Ref 19), austenite (Ref 14), ferrite-pearlite (Ref 16), virgin as well as stress-relieved martensite (Ref 17, 18), and the heterogeneous microstructure of the high-strength steel Fe-1.75%Ni-1.5%Cu-0.5%Mo-0.5%C made from partially prealloyed powders (DistaloyAE powders, made by HoeganaesAB, Sweden) (Ref 15). Density was varied within a narrow range (6.6 to 7.0 g/cm^3) to limit the study to those process conditions which combine reduced production costs (single press; single sintering; medium-high sintering temperature) and good mechanical properties. As a review of the results, only the materials listed in Table 1 (where chemical compositions and production schedules are reported) are considered here. Table 2 shows the main tensile properties, TRS values, and Weibull modulus of the same materials. Tensile tests for this investigation used a material testing system (MTS) testing machine with a crosshead speed of 0.5 mm/min and an extensometer with a gauge length of 25 mm, in accordance with ISO 2740. The total area under the load-deflection curves (the tensile fracture energy, TFE) was also calculated and included in Table 2, in order to evaluate the material toughness.

Table 1 Alloy compositions and sintering parameters

Material No.	Material description(a)	Density, g/cm^3	Sintering parameters(b) T_{sint}, °C	t_{sint}, min	Ref
1	Fe	6.6	1100	14	19
2	Fe	7.0	1100	14	19
3	Fe-0.3%C	6.6	1100	14	Present work
4	Fe-0.3%C	7.0	1100	14	Present work
5	Fe-0.25C-0.6P	6.8	1150	15	16
6	AISI 316L + 4%Cu	6.8	1180	30	14
7	DAE + 0.4%C	6.8	1150	20	17
8	DAE + 0.4%C AQ	6.8	1150	20	17
9	DAE + 0.4%C AQ + SR	6.8	1150	20	18
10	DAE + 0.4%C	7.0	1150	20	19

(a) The chemical composition of DAE is: Fe-4%Ni-1.5%Cu-0.5%Mo. AQ, as-quenched: austenitizing at 860 °C for 20 min and oil quenching at 65 °C; SR, stress relieved at 180 °C. (b) The atmosphere of sintering was endogas for the carbon-containing materials and 25%N$_2$-75%H$_2$ for material No. 6.

Table 2 Results of tensile and three-point bending tests

Material	$\sigma_{y0.1}$, MPa	σ_{UTS}, MPa	A%	TFE, J	TRS, MPa	m
1	110	150	4	4.2	378	36
2	150	220	6.3	6.7	506	41
3	148	210	4.2	5.6	417	39
4	180	290	6.4	9.7	497	45
5	330	423	5	12.8	800	55
6	256	320	3.2	4	820	40
7	340	540	2	8.2	1260	49
8	ND	860	<1	ND	1200	19
9	ND	980	<1	3.6	1400	31
10	430	700	2.5	13.2	1394	40

$\sigma_{y0.1}$, σ_{UTS}, and A% are the 0.1% offset tensile yield strength, the ultimate tensile strength, and the tensile elongation at fracture, respectively. TFE indicates the tensile fracture energy (ND, not determined).

The *m*-values in Table 2 show the good reliability of sintered ferrous alloys. The Weibull modulus ranges between 19 and 55; the lower value was determined for an as-quenched (not stress relieved) Fe-C alloy. In all other microstructural situations experimented, Weibull moduli higher than the above-mentioned threshold were determined. Consequently, porous sintered alloys, in general, have a mechanical reliability comparable to that of wrought steels. Porosity, while reducing mechanical properties, does not reduce the suitability of these materials for structural applications.

3. Interpretation of the Weibull Modulus on a Metallurgical Basis

The *m*-values can be interpreted on a metallurgical basis by considering the specificity of the mechanical behavior of these materials, which is derived from two main factors: (a) the presence of pores, with different morphological features; and (b) the use of particular types of powders and alloying elements, which often causes the formation, after sintering, of a complex and heterogeneous microstructure without any reference in the ingot metallurgy.

For example, this approach was accomplished with some Fe-C-P alloys, exploiting the possibility of obtaining different amounts of ferrite and pearlite with different contents of alloying elements by varying both the phosphorus and carbon in the chemical composition.

In Tables 1 and 2, only one alloy is reported (material No. 5). In the specific investigations (Ref 16, 22, 23), nine different alloys were produced with between 0.25 and 0.7 wt% C and between 0.45 and 0.8 wt% P. Both tensile and three-point bend tests were performed, and the fracture surfaces of the tested specimens were observed in a scanning electron microscope (SEM) to investigate the fracture mechanisms. The material microstructure was analyzed by the usual metallographic techniques, and the element distribution was highlighted by secondary ion mass spectroscopy. As a result of this investigation, a sequential correlation was determined among the chemical composition, microstructure, mechanical characteristics of the different microstructural constituents, the prevailing fracture mechanism, and the Weibull modulus. This correlation provides an interpretation of reliability on a metallurgical basis.

For example, at constant P content, *m* decreases as the carbon content increases (Fig. 1a). This results from the increase in pearlite content in the microstructure, which induces a prevailing cleavage fracture. Since cleavage fracture is promoted by the presence of pores (Ref 24), the different pore morphologies induce a scatter in the loads required to have fracture; as a consequence, the scatter of the mechanical test results is higher in the high carbon materials. On the other hand, for the low carbon alloys, plastic deformation in the ferrite reduces the effect of the great variability of the geometrical pore characteristics; thus, the statistical nature of the fracture process is reduced.

The variation of *m* with an increasing P content at constant C (Fig. 1b) presents a maximum for the intermediate value. Indeed, increasing the P content induced a monothonic increase in ferrite fraction and an increase in the activation of the sintering process with a corresponding increase in the pore roundness. The latter effect homogenizes the morphological pore characteristics and reduces the strain localization at the pore edges thus improving ductility. On the other hand, the influence of the increased ferrite content must be evaluated on the basis of the ferrite chemical characteristics. In fact, ferrite is present in two different forms: low-alloyed ferrite and P-enriched ferrite. Whereas low-alloyed ferrite gives rise to ductile fracture, P-enriched ferrite, which is strongly solution-hardened, fails by cleavage. By increasing the P content in the material, P ferrite prevails on the other, and fracture surfaces present an increasing fraction of cleavage areas. Consequently, the positive effect of sintering activation increases reliability up to a certain amount of phosphorus, above which microstructural embrittlement prevails.

In a previous investigation (Ref 16), a good correlation was found between the Weibull modulus for the Fe-C-P alloys and their elongation at fracture. This correlation is connected to the

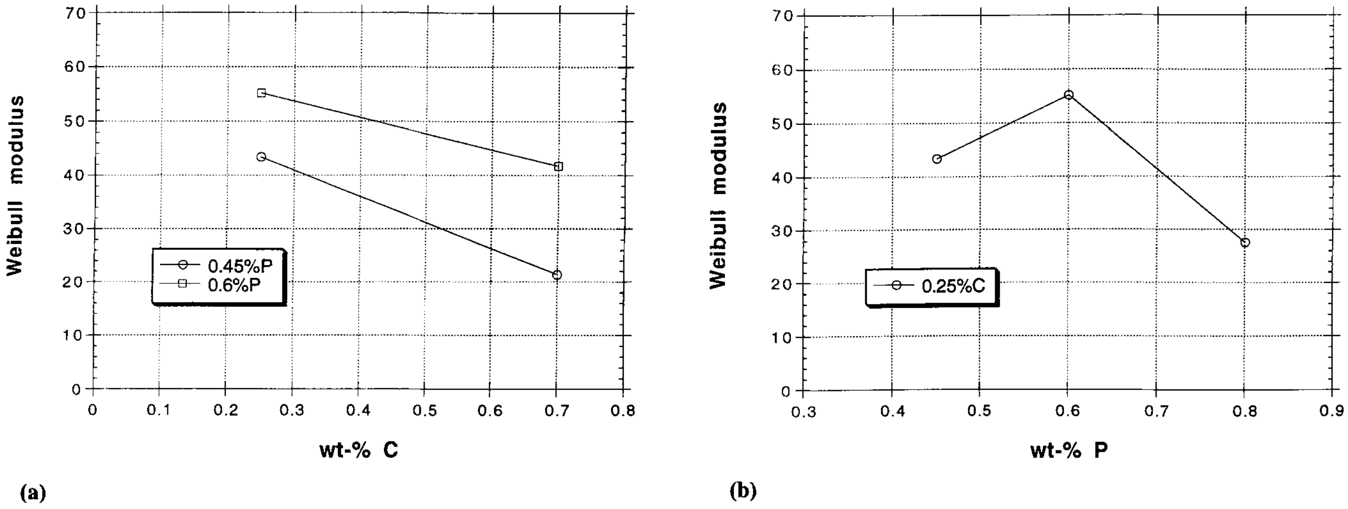

Fig. 1 Dependence of the Weibull modulus on (a) the C content for given P contents and (b) on the P content for a given C content (from Ref 16)

fact that the Weibull modulus usually increases when the material toughness is increased (Ref 25). (In the case of truly brittle materials, this is not always true according to Ref 26.) Within the same class of materials, the elongation at fracture can be roughly proportional to the material toughness. However, the correlation between the Weibull modulus and the elongation at fracture is different for the materials based on pure iron powders and for the Distaloy-type materials (Ref 17).

Figure 2 shows the experimental correlation between the Weibull modulus and the elongation at fracture for iron and the Fe-C alloys (materials No. 1 to 4) and for the Distaloy-type materials (materials No. 7 to 10). The figures further confirm that the Weibull modulus and elongation at fracture relationship exists only within a particular material class. The difference lies in the different stress-strain behavior of the two material classes, clearly highlighted by the tensile stress-strain curves in Fig. 3 relevant to material No. 2 (iron-based) and No. 10 (Distaloy-type). In particular, material No. 2 presents a stress-strain behavior characterized by the presence of a distinct yield point (and the same yields for materials No. 1 to 4). Material No. 10 shows a continuous yielding behavior (Ref 27) (like materials No. 7 to 10), which is due to the heterogeneous microstructure, characterized by the coexistence of hard (pearlite, bainite, martensite) and soft (ferrite) phases (Ref 28). See Fig. 4. Each material class is then characterized by a different deformation behavior: high flow stresses and low deformations for the Distaloy-type materials, low flow stresses and high deformations for the iron-based materials. This explains the existence of different correlations between the Weibull modulus and the elongation at fracture.

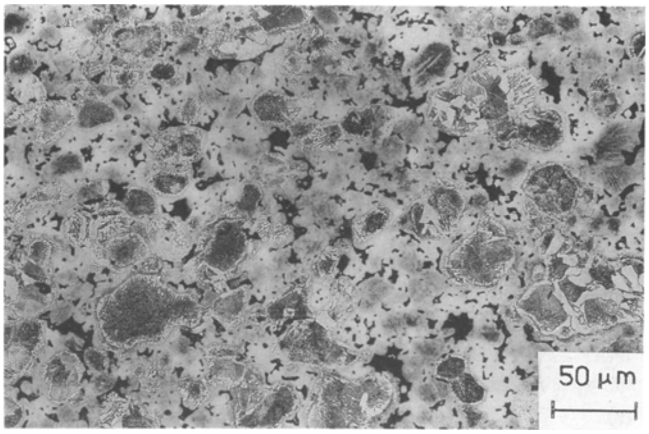

Fig. 2 Weibull modulus and elongation at fracture relationship for selected materials

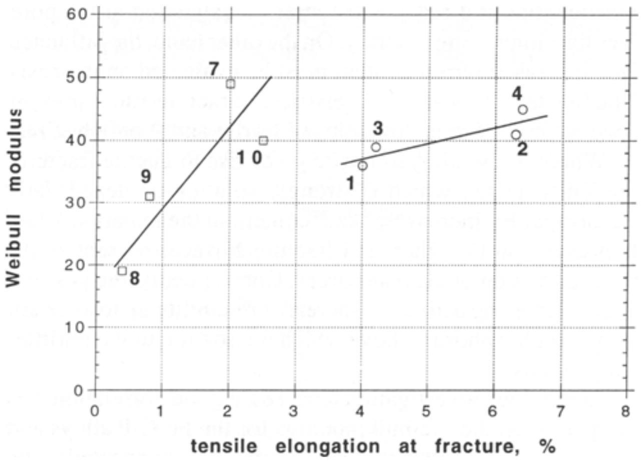

Fig. 3 Tensile engineering stress and strain curves for materials No. 2 and 10

A more general correlation is expected between the Weibull modulus and the tensile fracture energy, which is an integral value representative of the toughness of the materials. Figure 5 shows that all considered materials fall into an acceptable narrow scatter band with the sole exception of material No. 10. The anomalous behavior of material No. 10 is discussed here in the context of the analysis of the density influence on the mechanical reliability of P/M steels.

Another example concerning the need to consider the specificity of P/M materials in the study of their mechanical reliability is given by correlating *m* to density. In this case, both the pore morphology and matrix microstructure have to be properly considered.

Density is the most important parameter in P/M materials because of its positive effect on mechanical properties. For this reason, its influence on mechanical reliability was studied with reference to pure iron, Fe-0.3%C, and a Distaloy-based alloy with 0.5% C; that is, materials No. 1, 2, 3, 4, 7, and 10 in Tables 1 and 2. Figure 6 shows the Weibull moduli as a function of density. An opposite trend is apparent. Whereas *m* increases with density for pure iron and the iron carbon alloy, it decreases for the Distaloy-based material. In any case, high Weibull moduli were obtained.

The results here were also interpreted on a metallurgical basis by studying the fracture mechanisms. Pure iron presents ductile fracture at both density levels with an increase in the total fracture surface area with density (Ref 19). The same type of fracture is observed in the iron carbon alloy; see Fig. 7. In contrast, the Distaloy-based material presents a mixed type of fracture (dimpled areas and cleavages), and the fraction of cleavages increases as density is increased (Ref 19). See Fig. 8.

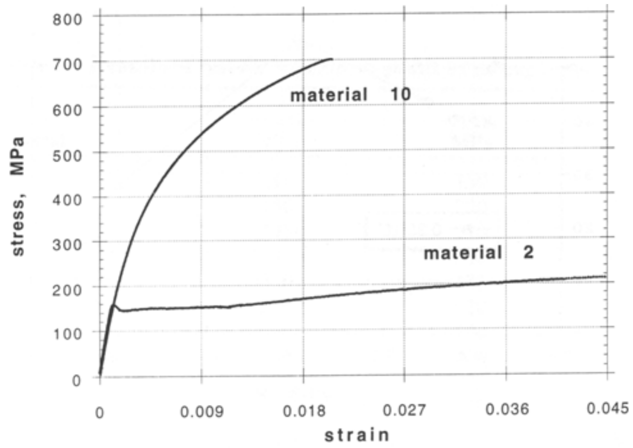

Fig. 4 Microstructure of material No. 7 showing the ferrite, pearlite, martensite, and upper bainite areas (3% nital etching)

This increased fraction of brittle areas is clearly responsible for the decrease in *m*, as schematized in Fig. 6.

These experimental findings are discussed in terms of the increased propagation of plastic deformation away from the neck area as density is increased. At low density, fracture is mainly localized at the necks; at high density, plastic deformation and fracture occur also in the bulk of the original powder grains. This evolution has different effects on the two types of materials because of their different microstructures. Iron is homogeneously ferritic, and Fe-0.3%C is constituted by ferrite and pearlite. The fracture is, in any case, ductile, and the increase in density increases the volume of material that undergoes plastic deformation before fracture. This has positive effects on toughness and reliability. In contrast, the deformation and fracture processes mainly occur in different microstructural constituents in the Distaloy-based material, depending on density. They are the ductile Fe-Ni phase (close to the neck zone) at low density and the less ductile pearlite or bainite (which constitute the bulk of the grains) at high density. Consequently, the increase in density increases mechanical strength but, at the same time, reduces reliability (Ref 19).

This method also explains the anomalous behavior of material No. 10 in Fig. 5, which has a lower Weibull modulus than that predicted by the tensile fracture energy (TFE). In the case of the Distaloy-type material, an increase in density increases TFE and reduces reliability. This is due to the different phenomena on which TFE and reliability are based. TFE is based on the plastic deformation preceding the onset of fracture. Reliability is based on the final fracture mechanism. The increase in density increases the amount of plastic deformation before fracture but, at the same time, induces the fracture process to involve less ductile constituents and then promotes cleavage fracture. Therefore, the results of mechanical tests are characterized by higher strength and elongation values and higher scatter.

The other materials fall into the same scatter band of Fig. 5 because, irrespective of density, they are all characterized es-

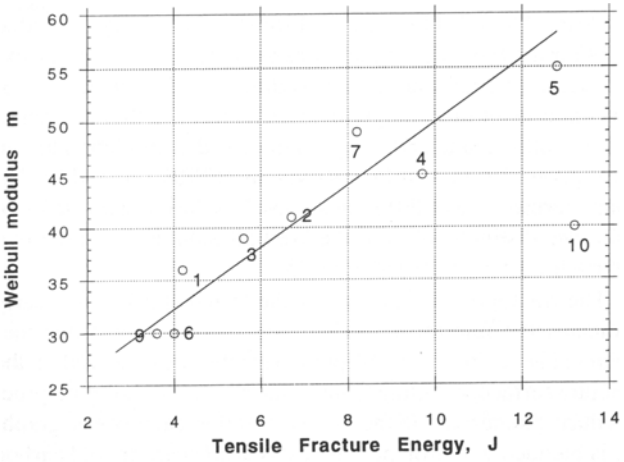

Fig. 5 Weibull modulus and tensile fracture energy relationship for the materials under study

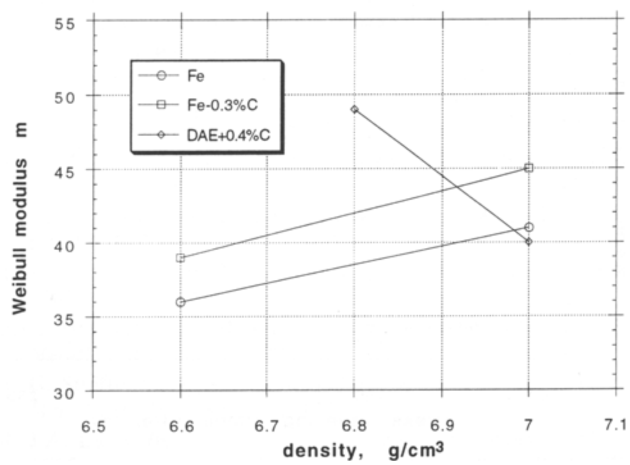

Fig. 6 Weibull modulus and density relationship for materials No. 1, 2, 3, 4, 7, and 10

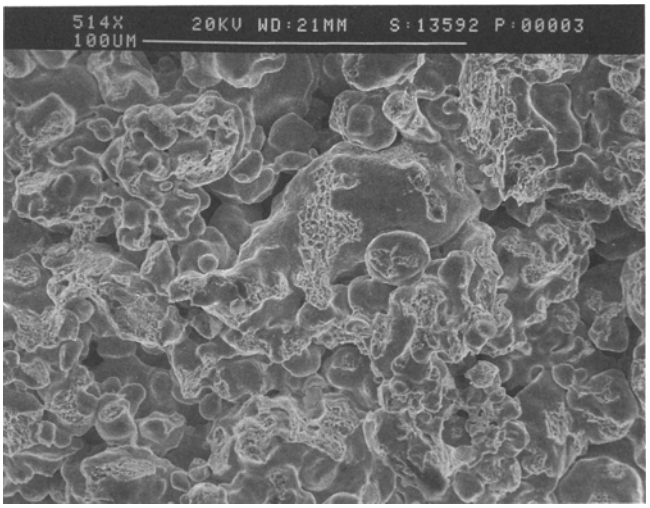

Fig. 7 SEM micrograph of the three-point bend fracture surface of material No. 3

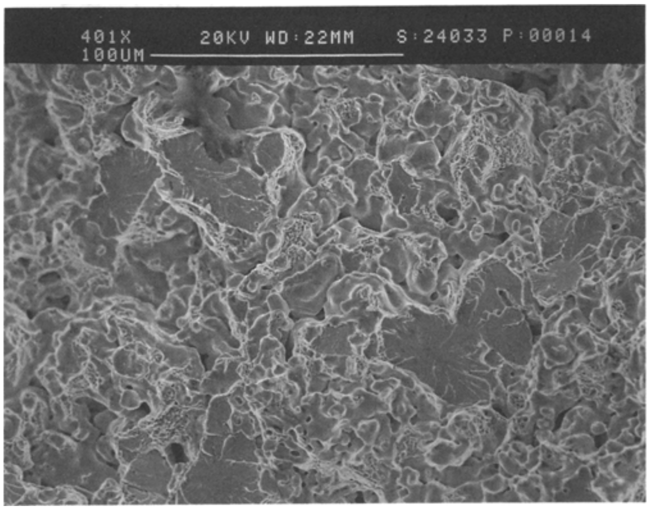

Fig. 8 SEM micrograph of the three-point bend fracture surface of material No. 7

sentially by the same deformation and fracture mechanism, which is ductile in nature although the flow behavior sometimes differs.

4. Application to Process Optimization and Control

The possibility of employing the Weibull approach for process optimization and control is investigated here. First, the optimization of the stress-relieving treatment for a carbon steel is considered.

Because of the effect of porosity on the mechanical properties of porous steels, the positive effect of stress-relieving treatment cannot be highlighted by the usual hardness, tensile, or impact tests, in particular for medium-high carbon steels. For example, Table 2 shows that elongation at fracture is negligible for materials No. 8 and 9, which differ only for the stress-relieving treatment. Similarly, the other above-mentioned mechanical parameters do not significantly change after stress relieving. Indeed, Table 2 shows a great and significant increase in *m* after treatment at 180 °C for 2 h. The Weibull analysis is then an objective demonstration of the necessity to stress relieve the as-quenched microstructure for sintered steels as well. This result constituted the basis for an extension of the experimentation aimed at the definition of the stress-relieving temperature (Ref 18). For this objective, the usual mechanical tests are not suitable; no significant variations in tensile strength, elongation, and impact energy are observed in the temperature range of 150 to 240 °C. As before, the Weibull modulus clearly indicates the influence of temperature, showing a transition from poor reliability to good reliability (Fig. 9). For the studied material, stress relieving must then be carried out at about 180 °C. Using the same approach, the correct treatment temperature was defined for other materials in air and oil.

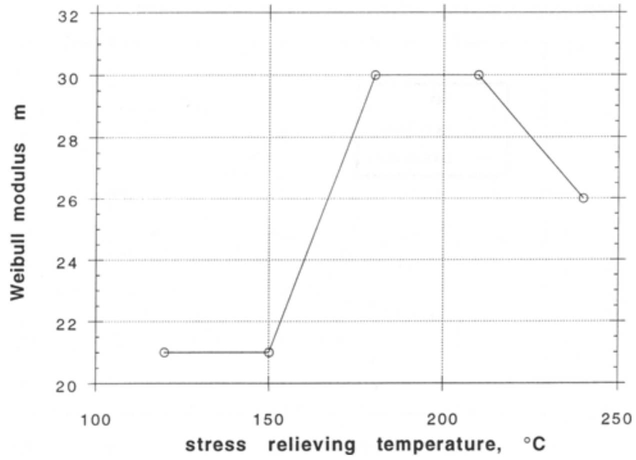

Fig. 9 Weibull modulus and stress-relieving temperature relationship for material No. 8

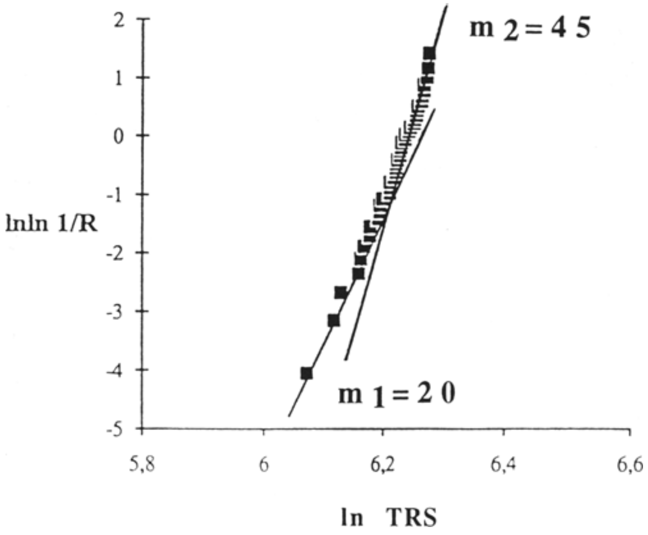

Fig. 10 Results of three-point bend test (TRS) for material Fe-1%C (density = 7.0 g/cm^3) presented as Weibull plot

Finally, another interesting application of the Weibull approach concerns the study of the mechanical reliability of an Fe-C steel with high (1 wt%) carbon content and a density of 7.0 g/cm^3, characterized by fairly typical tensile values ($\sigma_{y0.1}$ = 215 MPa, A% = 2, TFE = 5.5 J). From the data of Fig. 5, a value of 38 for the Weibull modulus was expected. However, the experimental Weibull modulus was dramatically lower, $m = 23$. The analysis of the strength distribution shows that this material is really characterized by a bimodal distribution. This, in turn, gives rise to a ln(ln 1/*R*) versus lnTRS curve (Fig. 10) characterized by two different slopes. It is thus possible to associate two distinct values of the Weibull modulus with the two distributions: $m_1 = 20$ and $m_2 = 45$.

The fractographic analysis of the tested specimens clearly indicates the microstructural reason for the anomalous mechanical behavior. Some graphite particles are observed on the fracture surface, resulting from an incomplete dissolution process during sintering. (In the usual technological process, graphite is blended with iron powder as a carbon carrier, and carbon diffusion into the iron lattice occurs during sintering.) The fractograph of Fig. 11 shows the occurrence of a crack adjacent to a graphite particle. A careful characterization of all the tested specimens showed that all the specimens with low strength values presented such anomalies, whereas the specimens with

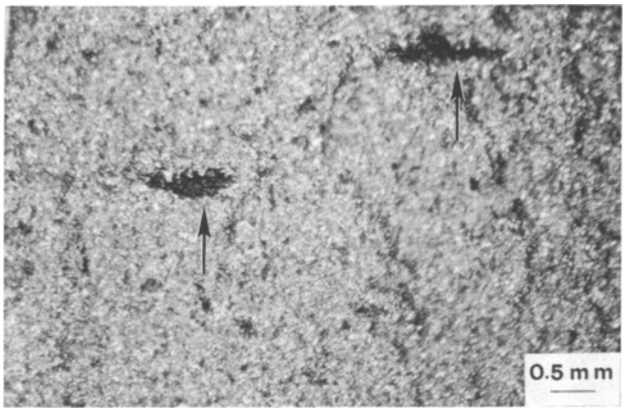

Fig. 11 Fracture surface of material Fe-1%C showing a crack adjacent to a graphite particle

higher strength values did not show the graphite cracks. Hence, the production process has to be adjusted in order to avoid such free graphite particles, which compromise the mechanical behavior of the material; that is, reduce its strength and increase its strength variability.

The Weibull analysis is a powerful method not only for the optimization of a material, but also for quality control. To support this suggestion, a Weibull analysis was carried out on real components too. For example, results obtained from radial crushing tests on bearings were comparable to those obtained from three-point bend tests on the same type of material sintered in the same batch. Thus, when geometric complexity does not introduce excessive density heterogeneities and a new source of strength scatter, it is not necessary to produce specific specimens for the evaluation of the Weibull modulus.

5. Conclusions

The main results of a five-year experimentation carried out on the mechanical reliability of porous P/M ferrous materials are given. Results include:

- In spite of porosity, sintered alloys, if correctly produced and heat treated, can have mechanical reliability comparable to that of conventional structural materials. For the selection of a suitable safety factor, they should not be considered as brittle.
- The Weibull modulus, which is the parameter representative of mechanical reliability, is correlated with the microstructural characteristics of the materials. Therefore, it can be interpreted on a metallurgical basis.
- The influence of density on mechanical reliability depends on the microstructural features of the different materials and requires a specific investigation for each material class.
- The Weibull approach proved to be a powerful tool for process optimization and control.

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