

## RELATIONSHIP BETWEEN PARTICLE SIZE AND MANUFACTURING PROCESSING AND SINTERED CHARACTERISTICS OF IRON POWDERS

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### Abstract

The effect of particle size distribution on physical properties of powder mixtures, of green compacts and of sintered samples has been studied. In the case of powder mixtures, the evaluated properties were flowability, apparent density, specific surface and compressibility. In green compacts porosity, roughness and green strength were evaluated, and in sintered samples grain size and transverse rupture strength were measured. In order to obtain samples with different average particle size, water atomized iron powders were sieved and separating it with sieves ranging from +44 to -150  $\mu\text{m}$

Flowability and compressibility decrease as average particle size does. On the opposite side, green strength and transverse rupture strength (TRS) increase as particle size diminishes. These effects were associated with changes in morphology and specific surface of the studied powder mixtures. Mathematical expressions relating average particle size with green strength, roughness and ultimate tensile strength were deduced from the experimental results. The results show that an appropriate selection of average particle size for the preparation of the powder mixture is useful in order to obtain samples with suitable physical properties.

**Keywords:** *particle size distribution, iron powder, apparent density, green strength and transverse rupture strength.*

### Resumen

En este trabajo se ha estudiado la influencia de la distribución de tamaños de partícula de polvo de hierro sobre propiedades de la mezcla tales como: fluencia, densidad aparente, superficie específica y compresibilidad, de los compactos en verde (porosidad y resistencia en verde) y finalmente de los compactos sinterizados (tamaño de grano metalográfico y la resistencia a la ruptura transversal (TRS)). Para llevar a cabo el desarrollo experimental se ha cribado polvo de hierro atomizado con agua, ASC 100.29 de Hoganas Co, separándolo en tamices que oscilan entre +44 $\mu\text{m}$  y -150 $\mu\text{m}$ . Los resultados revelan que las distribuciones de tamaños de partícula más pequeños empeoran la fluencia y la compresibilidad pero mejoran la resistencia en verde y la TRS. Lo anterior se asocia a las diferencias de morfología y de superficie específica de las mezclas estudiadas. Adicionalmente, se obtuvieron diferentes expresiones matemáticas que permiten establecer el comportamiento de la rugosidad de los compactos, la resistencia máxima y la resistencia en verde respecto al tamaño medio de partícula. Se concluye que una adecuada selección del tamaño de partícula para preparar las mezclas de polvo, puede ayudar a obtener una pieza sinterizada con las propiedades necesarias para su aplicación final ya que como se muestra en este estudio, ésta es una característica que afecta significativamente a cada una de las propiedades evaluadas.

**Palabras clave:** *distribución de tamaño de partícula, polvo de hierro, densidad aparente, resistencia en verde y resistencia a la ruptura transverse.*

## 1. Introduction

Powders commonly used in powder metallurgy are characterized by a given particle size distribution. This is selected as a function of the consolidation process and of the desired properties of the sintered sample [1]. The manufacturing process of a sample by powder metallurgy is conditioned by the properties of the initial powders. One of the most relevant properties is particle size distribution. Properties such as apparent density, flowability, specific area and compressibility depend on particle size distribution [2]. Additionally, in green compacts the properties such as porosity, roughness and green strength also depend on particle size distribution. The same is for transverse rupture strength (TRS) in sintered samples.

The effect of particle size distribution on properties of powder mixtures [3], of green compacts [4] and of sintered samples [5] has been previously reported. However, in-depth studies on this subject are still needed. It has been reported [6] that small particles generate a lot of inter-particle friction. This restricts the powders flow rate. Also, apparent density of small powders is higher than that shown by big powders of the same material. In the compaction process the use of small metallic particles originates problems during the pressing step. Some of these are: longer times to fill the matrix, damages due to wear in die and tooling elements, and as a result, a decrease in productivity [7]. These problems are the result of the increase in specific surface of the powders. Higher surface areas then increase friction between powder and matrix elements and inter-particle friction.

It has been reported that higher green strength of compacts is obtained with smaller particle size [8]. This is because a higher number of plastically deformed zones is obtained (physical union between or among particles). However, the opposite conclusion has been also reported [9]. According to these works, the morphology of smaller particles is more spherical and then weaker bonds are obtained.

The use of small particle size is based in the improvement of the mechanical properties of the sintered sample [10]. Higher surface area increases the number of bonds among the particles. Then, a more efficient solid state diffusion process is obtained due to the proximity of the powder particles.

Powders of small average particle diameter are used when high mechanical properties are desired. On the other hand, powders of larger sizes, are used to enhance mixing and compaction processes.

The mechanical properties of the sintered samples do not depend only upon particle size distribution. Morphology, structure and microporosity are also important. In this respect, Poquillon et al [11] have reported models which describe powder behavior during the compaction process of irregular and spherical powders.

Works previously published in this field are commonly related to properties of the powders mixture, of the green

compact or of sintered sample properties. However, each step in the production process is commonly studied independently. To our knowledge, in-depth studies of the effect of particle size distribution on the properties of green compacts and sintered samples have not been published. So, the main purpose of this work is to describe how particle size distribution affects some of the most important variables in the powder metallurgy process.

## 2. Experimental Procedure

Water atomized iron powder, ASC 100.29 Hoganas Co., was used. An original lot of this powder was screened into five fractions using Tyler certified U.S. series sieves according to ASTM-B214-76 [12], as follows: 44, +44 to -74, +74 to -105, +105 to -150 and +150  $\mu\text{m}$ . Sieves were standardized. Powder morphology was characterized using scanning electron microscopy (JEOL-6300).

Particle size distribution was measured by laser diffraction (Beckman Coulter LS 13320). Specific surface area was measured by the BET method [13] (Micrometrics ASAP 2405 N Automatic Nitrogen Adsorption Pore Analyzer). Also, the specific surface,  $S_e$  ( $\text{m}^2/\text{g}$ ), was evaluated with the following

expression:  $S_e = \frac{6}{D_m \cdot \rho_t}$  where  $D_{m,0.5}$  is the average particle

diameter and  $\bar{\rho}_t$  is the theoretical or crystallographic density ( $\text{g}/\text{cm}^3$ ) of the powder mixture. In order to evaluate the effect of adding a pressing lubricant, each powder sample was mixed with 0.8 wt% zinc stearate. A laboratory type "V" mixer was used at 50 r.p.m. during 20 min. In samples with and without lubricant, apparent density (MPIF 04-B212) [14] and flow rate (MPIF 04-B212) [14] were measured. In samples with lubricant compressibility was also measured MPIF 04-B212) [14].

Specimens used in TRS and green strength tests were produced using a laboratory press; with 60 metric tones of maximum capacity. A tungsten carbide matrix was used. Uniaxial load was applied on the sample upper face. Samples with transverse rectangular section were obtained in accordance with MPIF-15 (ASTM B312). Density of the green compacts was  $6.8 \text{ g}/\text{cm}^3$ . Green strength was evaluated using the three points bending test. A Shimadzu 600476-04 equipment was used. Density of the green compacts was evaluated by Archimedes method. Roughness,  $R_a$ , was measured using Mitutoyo SurfTest 301 equipment.

Green samples were sintered in a belt furnace at  $1120^\circ\text{C}$  during 20 minutes, under a dissociated ammonia atmosphere. Sintered samples were etched with 2% of nital and grain size was determined according to ASTM112-88 [12]. TRS was measured in accordance with ASTM B528 using an equipment Instron 8802. Green strength was determined according to MPFI 04-B212 [14]. TRS and green strength were cal

culated with the following expression:  $\sigma = \frac{3PL}{2Wt^2}$ , where

P is the maximum or peak load, L distance, W is the width, and t is the sample thickness.

### 3. Results and Discussion

In figure 1 particle size distributions of the different powder fractions are shown. The lines show a correct sieving of the original powders. Unimodal distributions with narrow bands were obtained, so the average particle size can be considered as representative of each fraction.

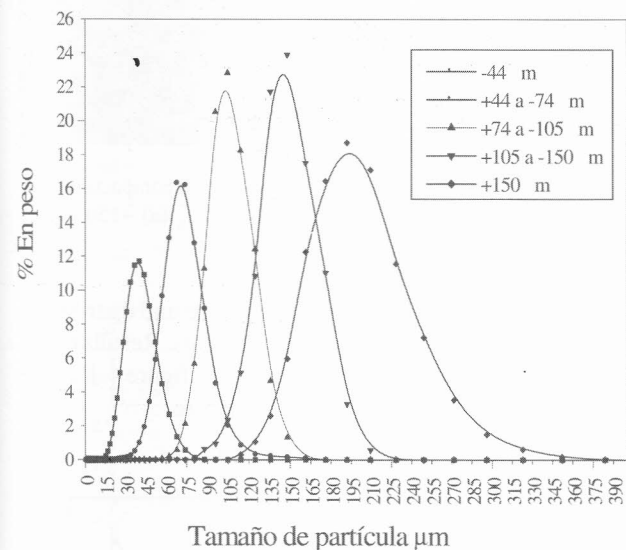


Fig. 1. Particle size distribution of sieved powder.

In table 1 average particle size and specific surface (experimental and theoretical) of the iron powder fractions are reported. It can be observed in this table, the specific surface,  $S_e$ , diminishes as average particle size increases; however, this decrease is also affected by particle morphology and microporosity.

Table 1. Physical properties of sieved powder.

Fraction (μm)	$D_{m,0.5}$ (μm)	$S_e$ (BET) (m <sup>2</sup> /g)	$S_{e,th}$ (m <sup>2</sup> /g)
-44	33,9	0.287	0.0234
+44 a -74	63,1	0.162	0.0126
+74 -105	95,8	0.113	0.0083
+105 a -150	140,1	0.098	0.0057
+150	188,7	0.055	0.0042

When the values of theoretical and measured specific surfaces are compared, differences in one order of magnitude are observed. This could be caused by particle morphology and internal microporosity. Smaller disagreements were found in fractions with smaller average particle size. In

the case of fraction -44 μm the ratio theoretical to experimental specific surface,  $S_{e,th}/S_e$ , was 12.26. It is in agreement with the nearly spherical shape of this fraction; as shown in figure 2 (a). On the other hand, in the fraction +74 to -105 μm the ratio  $S_{e,th}/S_e$  was 13.61. This higher ratio can be explained in terms of the more irregular morphology of the iron particles; as shown in figure 2 (d). In figure 2 it can be observed that all the powder fractions have irregular morphology. It could have been originated by the coalescence of almost spherical particles with several sizes.

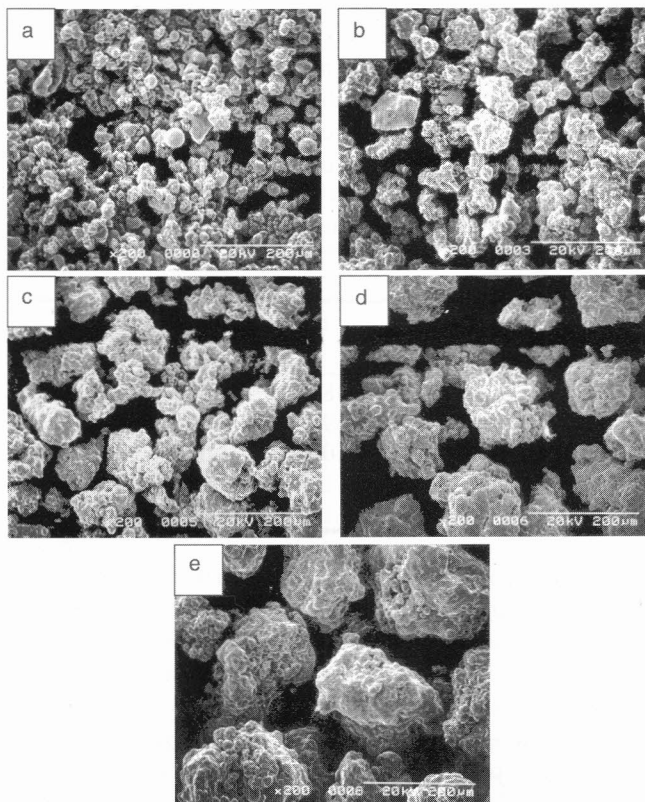


Fig. 2. SEM image of the powder morphology (a) -44 μm, (b) +44 a -74 μm, (c) +74 a -105 μm, (d) +105 a -150 μm (e) +150 μm.

In table 2, fluency and apparent density values of the powder fractions without lubricant, are reported. In all the samples small variations of fluency with average particle diameter were found.

Table 2. Apparent density and flow rate of the powder fractions without lubricant.

Fraction (μm)	Flow rate (s/50g)	$\pm\sigma$	$d_a$ (g/cm <sup>3</sup> )	$\pm\sigma$
-44	26,28	0.3327	2,90	0.0110
+44 a -74	24,53	0.1313	2,77	0.0015
+74 -105	28,13	0.2280	2,79	0.0063
+105 a -150	29,00	0.0670	2,86	0.0189
+150	27,29	0.1738	2,89	0.0548

As it can be seen in table 2, the apparent density diminishes when the particle size is decreases, due to internal friction increases. Exceptionally, the -44 fraction is more dense because it have spherical particles, and the internal friction is minor.

On the other hand, flow rate is not only affected by the number of contacts between particles. It also involves other factors such as: ordering, spatial distribution and morphology of the powder particles.

Only in the fraction -44  $\mu\text{m}$  a noticeable change in flow rate was found after lubricant was added. Lubricant binders small powder particles and acts as a glue restricting particle sliding. In the other fractions no strong effect of lubricant on flow rate was found, once the standard deviation is considered. Only a modest improvement in flow rate was found as a consequence of the lower inter-particle friction.

Table 3. Apparent density and fluency of the powder fractions with lubricant.

Fraction ( $\mu\text{m}$ )	Fluency (s/50g)	$\pm \sigma$	$d_a$ ( $\text{g}/\text{cm}^3$ )	$\pm \sigma$
-44	55,51	2.3805	3,32	0.0103
+44 a-74	23,23	0.1997	3,13	0.0190
+74 -105	26,99	0.1068	3,00	0.0104
+105 a-150	28,43	0.1173	3,02	0.0060
+150	27,56	0.1143	2,99	0.0081

It can be observed in the table 3 that in the fractions with lubricant, apparent density reduced slightly as average diameter increased. It could be caused by a better arrangement due to a smaller range of size distribution.

Compressibility of the powder fractions is reported in table 4. Compressibility increases with average particle diameter. Bigger particles have less contact surface than smaller ones and then less inter-particle friction is found in the first case. This improves particle mobility in the first reordering step [17] and particles packing increases.

Table 4. Compressibility of the powder fractions with lubricant.

P (MPa)	$d$ ( $\text{g}/\text{cm}^3$ )				
	-44 $\mu\text{m}$	44 to74 $\mu\text{m}$	74 to 105 $\mu\text{m}$	105 to150 $\mu\text{m}$	+150 $\mu\text{m}$
200	5.71	5.82	5.81	5.90	5.85
400	6.62	6.74	6.79	6.80	6.82
600	7.05	7.13	7.16	7.18	7.19
800	7.22	7.27	7.30	7.32	7.34

Samples of the different powder fractions were compacted to a density of  $6.8 \text{ g}/\text{cm}^3$ . Which all have the same porosity. However, porous morphology, size and distribution changed with average particle diameter. This can be observed in the micrographs shown in figure 3. These differences in poros-

ity are a consequence of the changes in grain size distribution in each powder fraction. Porosity is partially created by air or lubricant occlusion of the compacted powder particles. Porous size increases with particle size, but porous number decreases.

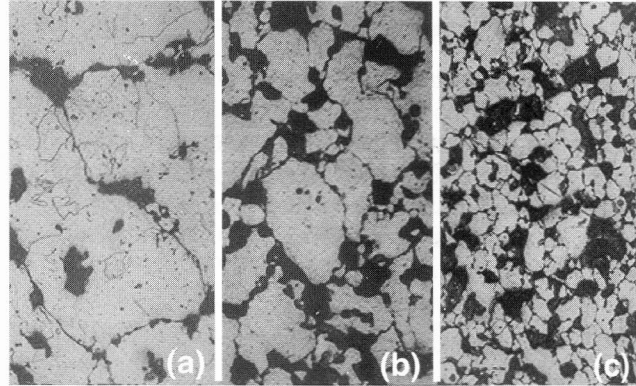


Fig. 3. Micrographs of transverse sections of green compacts pressed at  $6.8 \text{ g}/\text{cm}^3$  density, corresponding to fractions: (a) +150  $\mu\text{m}$ , (b) +74 a 105  $\mu\text{m}$  y (c) -44  $\mu\text{m}$ . nital 2%. X100.

Differences in porous morphology, size and distribution were quantified by roughness measurements. Results of this parameter,  $R_a$ , of each fraction are shown in figure 4. It can be observed that  $R_a$  increases with particle size.

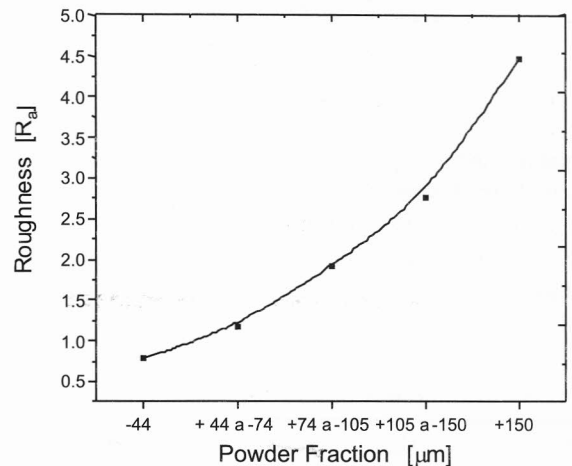


Fig. 4. Measured roughness in each powder fraction.

Results of green strength measurements are shown in figure 5 (a). Green strength reaches a maximum value at intermediate particle sizes due to the effect of particle morphology and number of bonds (contact zones during compaction). In fractions with small particle size a more spherical shape causes less interlocking between particles. In fractions with large particle size a fewer average number of mechanical bonds between particles produces weaker samples.

Then, the maximum in green strength is observed at intermediate particle sizes. In this case the conditions of particle morphology and number of bonds are more favorable to obtain higher mechanical strength [18]. It can also be observed that the maximum in green strength was measured in the sample formed by compaction of the powder fraction having the most irregular shape.

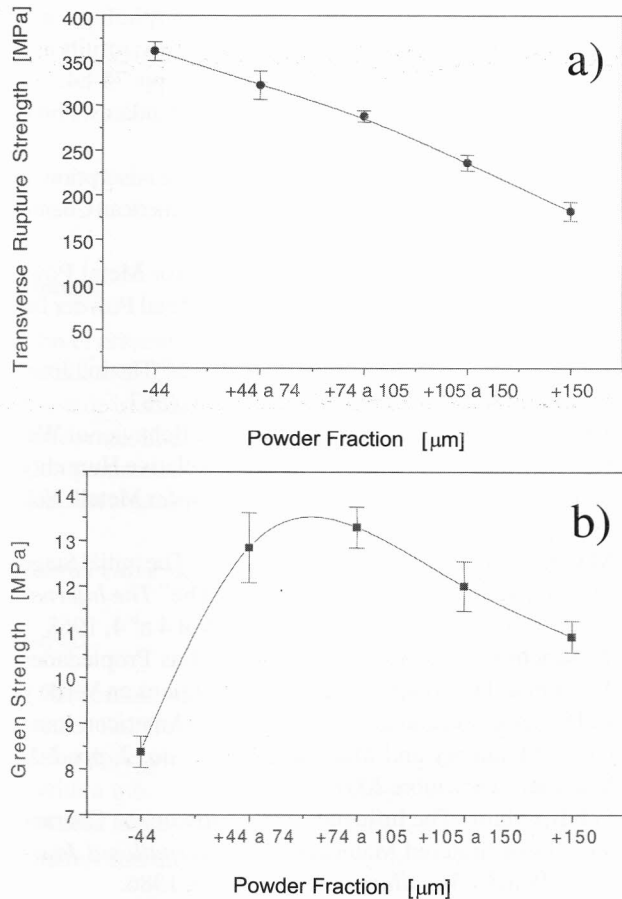


Fig. 5. (a) Green strength and (b) transverse rupture strength corresponding to the different powder fractions.

Transverse rupture strength decreases as powder diameter increases; as shown in figure 5 (b). Two factors could have originated this behavior. First, larger particles originate larger pores and these act as stress raisers [19]. Second, specific surface increases as particle diameter decreases; therefore, the number of metallurgic bonds increases when small particles are sintered.

Average grain sizes measured in sintered samples, corresponding to each powder fraction are shown in figure 6(a). As expected, average grain size increased with particle size.

The plot of transverse rupture strength vs. (average grain size)<sup>-1/2</sup> is shown in figure 6 (b). An almost perfect fit to a linear relationship,  $r^2=0.995$ , was found. The corresponding Hall-Petch [20] relationship can be expressed as TRS (MPa) =  $-48.9 + 1625.9 d^{-1/2}$ ; where  $d$  is expressed in

micrometers. From figure 6 it can be concluded that the mechanical properties of the sintered samples can be controlled with a careful selection of the initial powders size.

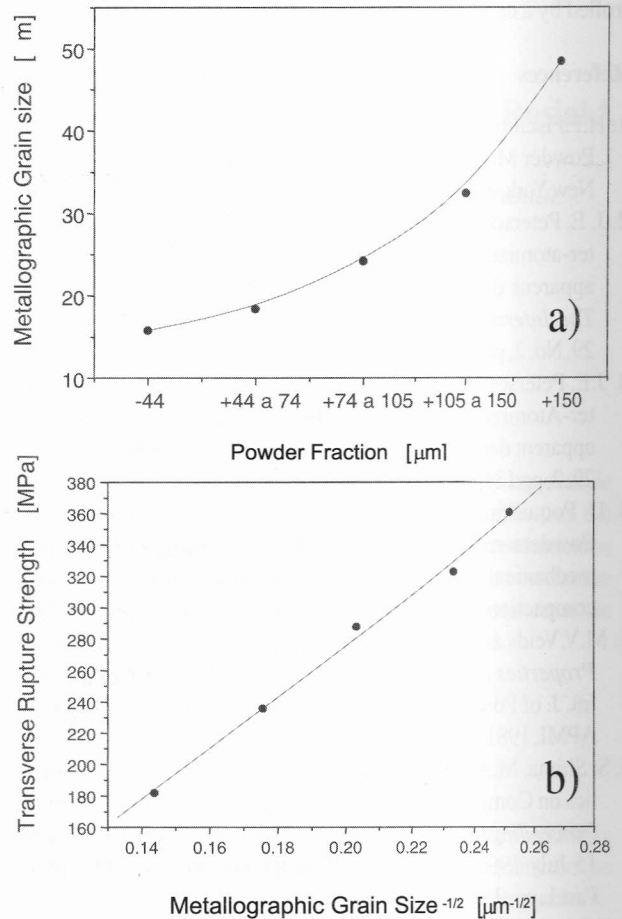


Fig. 6. Relation between metallographic grain size ( $T_g$ ) and (a) particle size (powder fraction) and (b) Transverse Rupture Strength.

#### 4. Conclusions

Apparent density, flowability and compressibility of powder mixtures are characteristics of these materials which depend upon of (1) particle morphology and (2) particle size distribution. However, these properties can also be affected by the presence of lubricant.

Porosity (percentage, distribution and size) of P/M compacts depends upon of the initial powder size. Small powder particles produce a high quantity of "small" pores distributed through the entire compact. Powder particles with a large particle size produces a small number of pores, heterogeneously distributed in the sample and of "large" size.

Green strength reached a maximum value in particles of intermediate size. This was produced by the combination of particle morphology and number of bonds effects in the pressed samples.

Transverse rupture strength decreases as grain size decreases. The variation of transverse rupture strength with

grain size to the  $-1/2$  power, can be expressed with a Hall-Petch relationship. Also, average grain size depends upon particle size distribution in the starting material. Then, the mechanical properties of the sintered sample can be controlled by a careful selection of the particles size range.

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