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September 21, 1945.

R E P O R T

of the

ORE DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 1936.

Metallurgical Examination of Iron Powder.

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Origin of Material and Object of Investigation:

In March, 1945, the Consolidated Mining and Smelting Company of Canada Limited, Trail, B.C., per F. H. Chapman, Secretary, Research and Development Board, submitted a sample of iron powder, reported to have been prepared by gas reduction of roasted specially purified iron sulphide concentrate.

It was requested that this powder be examined to determine its suitability for powder metallurgy fabrication processes.

Physical Characteristics of the Powder:

Chemical Analysis -

Chemical analyses are shown in Table I. The hydrogen loss is considered as an indication of the oxide content and was determined by heating approximately eight grams of the powder in an alundum boat at 1000°C. for one hour in a hydrogen atmosphere, cooling in the furnace to room temperature, and then weighing the sample.

All other determinations were carried out by standard methods of iron and steel analysis.

TABLE I. - Chemical Analysis.

	<u>Per Cent</u>
Total iron -	97.38
Silica -	0.26
Manganese -	0.12
Sulphur -	0.025
Phosphorus -	0.009
Hydrogen loss -	1.99

Screen Analysis -

Screen analyses were made with standard Tyler 8-inch screens and a Ro-Tap machine. The screening time was 10 minutes. A 500-gram sample was used.

TABLE II. - Screen Analysis.

	<u>+100</u>	<u>+150</u>	<u>+200</u>	<u>+270</u>	<u>+325</u>	<u>-325</u>
Iron Powder -	0	1.9	27.3	22.8	27.8	20.2

Flow Rate -

The flow rate of a powder is determined as the length of time, in seconds, required for 50 grams to fall freely through the orifice of a standard flowmeter. A flowmeter consists of a funnel the sides of which form a 60° angle and at the base of which is an orifice 0.10 inch in diameter.

The result for this powder is shown in Table III.

(Physical Characteristics of the Powder, cont'd) -

Apparent Density -

The Scott volumeter is used to measure the apparent density of powders. In this instrument the powder falls down, a baffle tower under a constant head and is collected at the bottom in a one-cubic-inch container. The weight of this volume of powder is determined. The apparent density is recorded in Table III.

Specific Surface -

The specific surface of a powder is defined as the surface area of powder particles, expressed in square centimetres per gram. It was determined by a Bowen fineness meter, whereby the air flow through a calibrated orifice is compared with that through a bed of powder. The result is given in Table III.

TABLE III.

	<u>Flow Rate</u>	<u>App. Density</u>	<u>Specific Surface</u>
Iron Powder -	No flow.	25.7 gm./cu.in.	1210 sq.cm./gm.

Mechanical Properties of Compacts:

To investigate the pressing characteristics, the powder was mixed with 1 per cent stearate as lubricant and pressed in a die which had been designed to give a compact of 1 square inch cross-section. The well depth of the die was 2 inches and the height of the compacts was kept as close to $\frac{1}{8}$ inch as possible, thus the resultant volume was approximately $\frac{1}{8}$ cubic inch. A forming pressure of 40,000 p.s.i. was used and was applied on one side of compact only. Due to the low apparent density of this powder, sufficient sample to form a compact with $\frac{1}{8}$ inch height under a pressure of 100,000 p.s.i. could not be placed in the die.

(Continued on next page)

(Mechanical Properties of Compacts, cont'd) -

The compressibility of powders is expressed in terms of

(a) the compressibility density, i.e., the density in grams per cubic inch of a compact formed at a specified pressure, in this case, 40,000 p.s.i.,

or

(b) the compressibility ratio, which is $\frac{\text{volume before pressing}}{\text{volume after pressing}}$

for any given pressure.

Both were measured and are recorded in Table IV.

The green compacts were sintered in a dry, purified hydrogen atmosphere at 1000° C. for one hour and cooled in the furnace. Four tensile test specimens, 0.188 inch in diameter and of $\frac{5}{8}$ inch gauge length, were machined from the sintered compacts. The ultimate strengths after pressing at 40,000 p.s.i. are shown in Table IV.

TABLE IV.

<u>Iron Compact</u>	<u>Compressibility</u>		<u>Tensile</u> <u>Strength,</u> <u>p.s.i.</u>
	<u>Density,</u> <u>gm./cu.in.</u>	<u>Ratio</u>	
At 40,000 p.s.i.* =	81.7	3.20	15,800
	81.7	3.20	15,700
	81.7	3.20	16,800
	81.7	3.20	16,400

* Powder was too bulky to press a suitable compact at higher pressures.

The per cent porosity of the compact and the per cent shrinkage due to sintering are shown in Table V.

TABLE V.

<u>Shrinkage,</u> <u>per cent</u>	<u>Porosity,</u> <u>per cent</u>
3	29.8

Microscopic Examination:

The shape of the powder particles was determined by examination at 75 diameters under a binocular microscope.

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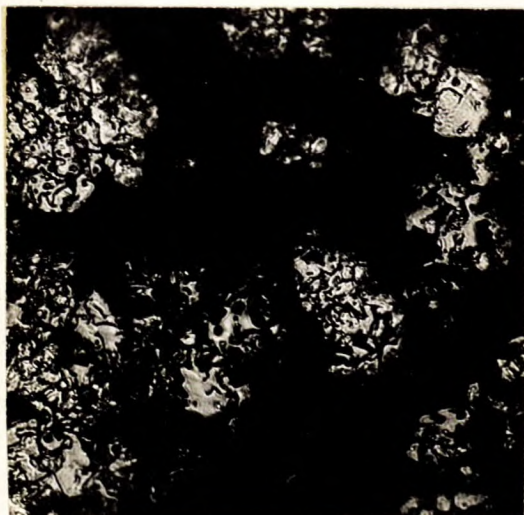
(Microscopic Examination, cont'd) -

Both large and small sizes tended to be globular in shape and were quite porous. No attempt was made to classify the particle size distribution by microscopic methods.

For further examination a sample was mounted in bakelite, polished, and etched with 2 per cent nital. As shown in Figure 1, a photomicrograph at X500, the particles are quite porous and contain a relatively high percentage of iron oxide (dark grey constituent) within the pores.

The microstructure of one of the sintered compacts is shown in Figure 2, a photomicrograph at X250.

Figure 1.



X500, etched in
2 per cent nital.

POWDER PARTICLES.

Figure 2.



X200, etched in
2 per cent nital.

COMPACT PRESSED AT 40,000 P.S.I. AND
SINTERED AT 1000° C. FOR 1 HOUR.

Large dark grey area is particle of iron
oxide, the other dark areas are voids.

Discussion:

The characteristics of an iron powder which have a predominant influence on its suitability for any powder metallurgy fabrication process are: (1) chemical composition, (2) particle shape and size distribution.

Although it is not possible to evaluate an iron powder solely on the basis of chemical analysis, the usual range for powders of this class (97% Fe +) is as follows:

	<u>Per Cent</u>
Total iron -	95-98
Total carbon -	0.30 max.
Hydrogen loss -	1.50 max.
Silica -	0.50 max.
Phosphorus -	0.020 max.
Sulphur -	0.025

With the exception of the hydrogen loss, the chemical analysis as shown in Table I of this report is within the above range. This hydrogen loss of 1.99, substantiated by the findings of microscopic examination, indicates a relatively high iron oxide content.

The particle shape of iron powders is generally classified as either dendritic, granular, globular, or flaky, with particular reference being made to the degree of porosity. These variations in shape, together with the particle size distribution, have a considerable bearing on such properties as apparent density, compressibility, specific surface, and also the pressure and length of heat treatment required to give desired characteristics in the final product. For example, porous particles and a large proportion of fines in a powder result in low apparent densities and poor flow properties. As opposed to this, powders with fairly dense, more or less rounded particles require higher compacting pressures and longer heat treatment cycles to produce the required mechanical strength.

The powder under consideration, which is very

(Discussion, cont'd) -

porous and whose particle size distribution contains more than the usual number of fines, has properties which are in agreement with these characteristics. The apparent density of 1.57 is low as compared with 1.9 to 2.2 for presently marketed powders of this class. As a result, the powder is bulky and could not easily be handled in dies designed for mass production. The poor flow properties are objectionable for the same reason. Since these properties can be controlled to some extent by re-adjustment of the particle size distribution, the following screen analysis is given as a typical specification, although there is considerable variation in such specifications, depending on the ultimate purpose for which the powder is required.

	<u>Per Cent</u>
On 60 mesh -	6 max.
" 100 "	17-20
" 150 "	12-16
" 200 "	15-20
" 250 "	5-8
" 325 "	10-14
Through 325 "	25-32

A comparison of this specification with the screen analysis given in Table II will show that the sample has a much higher percentage in the -200 mesh fraction.

The pressing and sintering properties of the powder also reflect the porous nature of the particles and the large proportion of fine mesh size--i.e., the density of the pressed compacts is low--and the compressive ratio is higher than usually desired in this type of powder. The tensile strength, however, is comparable to that obtained from commercial powders of this class, pressed and sintered at the same time.

Generally, the chemical composition of this powder is in good agreement with that of currently marketed powders, with the exception of the high iron oxide content.

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(Discussion, cont'd) -

It is felt, however, that too much emphasis should not be placed on the results of pressing and sintering tests shown in this report. Tests on a larger sample with a more suitable apparent density and flow properties would give more conclusive results.

It is possible that the low density and flow characteristics of this powder could be improved by a pre-conditioning of the powder after it has left the furnace, by simply placing it in a ball or rod mill and subjecting it to a rolling action to partially compress the porous particles.

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IHM:GTS:LB.