2.

PRODUCTION OF IRON AND STEEL POWDERS

This chapter presents a short history of iron powder and a brief account of how world production and consumption of iron powder have developed since 1965. It describes in detail two production methods which together account for more than 90% of today’s world production of iron and steel powders.
2. PRODUCTION OF IRON AND STEEL POWDERS

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2.1 Introduction

Iron and steel powders for the manufacturing of sintered structural components (including sintered porous bushings) are produced in many parts of the world. The worldwide consumption of such powders has been growing increasingly fast over the last three decades and exceeded 550 000 metric tons by the end of 1995. See Fig. 2.1.

Figure 2.1  World-wide consumption and usage of iron powder over the period from 1965 to 1995. (A) Low alloyed iron powders; (B) iron powders for high density sintered parts; (C) iron powders for low and medium density sintered parts. Low Density: 5.5 - 6.6 g/cm³, Medium Density: 6.7 - 7.0 g/cm³, High Density: > 7.0 g/cm³.
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Over the last thirty years, the quality of iron and steel powders has been continuously improved and the spectrum of available grades has been widened. During the same period, compacting- and sintering-techniques have become more and more sophisticated. This development has lead to a substantially widened range of applications for sintered iron and steel parts.

Table 2.1 World-wide Usage of Iron Powder for Sintered Parts

<table>
<thead>
<tr>
<th>Year</th>
<th>Unalloyed Iron Powders</th>
<th>Low-alloyed Iron Powders</th>
<th>All Iron Powders</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>compact densities 5.5 to 7.0 g/cm³</td>
<td>compact densities &gt; 7.0 g/cm³</td>
<td>compact densities &gt; 6.7 g/cm³</td>
</tr>
<tr>
<td></td>
<td>tons</td>
<td>%</td>
<td>tons</td>
</tr>
<tr>
<td>1965</td>
<td>35 000</td>
<td>100.0</td>
<td>-</td>
</tr>
<tr>
<td>1975</td>
<td>120 000</td>
<td>92.3</td>
<td>10 000</td>
</tr>
<tr>
<td>1985</td>
<td>180 000</td>
<td>70.6</td>
<td>50 000</td>
</tr>
<tr>
<td>1995</td>
<td>300 000</td>
<td>53.6</td>
<td>200 000</td>
</tr>
</tbody>
</table>

From Table 2.1, it can be seen that in 1965 iron powders were used almost exclusively for low- and medium-density applications, i.e. for parts having pressed densities from 5.5 to 7.0g/cm³. First after about 1970, increasing quantities of iron powders were used for high-density applications, i.e. for parts having pressed densities higher than 7.0g/cm³. Between 1975 and 1985, low-alloyed iron powders appeared on the marked and have since been used in growing quantities for medium- and high-density applications, i.e. for parts having densities higher than 6.7g/cm³.
2.1 INTRODUCTION

It must be mentioned that, at present (1996), the worldwide production capacity of iron and steel powders is considerably larger than the consumption. Thus, there is no risk of shortage for many years to come. See Table 2.2.

At present there are two basically different production methods which together account for more than 90% of the world production of iron and steel powders, viz. the Höganäs sponge-iron process and the water-atomizing process. The former process is based on reduction of iron ore, yielding a highly porous sponge-iron which subsequently is comminuted to powder. The latter process is based on atomization of a stream of liquid iron (or steel) by means of a jet of pressurized water. Both processes will be described in detail further below.

In the manufacturing of sintered parts, iron powders are always used admixed with a small amount of lubricant in powder form in order to minimize the friction in the compacting tool. In many cases, they are also blended with alloying elements in powder form, like graphite, copper, nickel, molybdenum and others (in order to achieve increased strength properties).

Since powder blends tend to segregate when transported and handled, Höganäs AB has developed special blending processes in which the alloying additives are safely bound to the iron powder particles. These processes have become known under the names Distaloy and Starmix, and are also treated in detail further below.

Table 2.2 Production Capacity and Consumption 1995 of Iron Powder for sintered parts and all other applications

<table>
<thead>
<tr>
<th></th>
<th>Production Capacity tons</th>
<th>Consumption tons</th>
</tr>
</thead>
<tbody>
<tr>
<td>Europe</td>
<td>400 000</td>
<td>160 000</td>
</tr>
<tr>
<td>Americas</td>
<td>600 000</td>
<td>310 000</td>
</tr>
<tr>
<td>Asia</td>
<td>300 000</td>
<td>200 000</td>
</tr>
<tr>
<td>World Total</td>
<td>1 300 000</td>
<td>670 000</td>
</tr>
</tbody>
</table>

It must be mentioned that, at present (1996), the worldwide production capacity of iron and steel powders is considerably larger than the consumption. Thus, there is no risk of shortage for many years to come. See Table 2.2.
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2.2 Short history of iron powder

Industrial production of iron powder started in 1937 on the incentive of General Motors Corporation in the USA. Höganäs was active since 1922 in producing a high-quality sponge iron to be used by the Swedish steel industry as high-purity melting stock for the production of special steels like tool-steel and stainless steel.

Initial tests in 1937 showed that, owing to its high porosity, the sponge iron produced by Höganäs could readily be comminuted to iron powder. Production of iron powder started on a small scale at Höganäs in Sweden between 1937 and 1939, and regular shipments were made to the USA.

These early grades of sponge iron powder were unannealed, containing up to 2% of reducible oxygen (H₂-loss) and up to 0.15% of carbon. Consequently, their compressibility was poor, and the reduction of residual oxides had to take place in the sintering process under consumption of carbon present in the compacts. Under such conditions, it was impossible to use carbon as an alloying element. The bulk density (apparent density) of these powder grades was relatively low, i.e. 2.2 to 2.4 g/cm³.

After 1940, Höganäs introduced an annealing procedure in which the crude iron powder was heated under dissociated ammonia (75% H₂ + 25% N₂) resulting in considerably lower contents of residual oxygen and carbon and yielding a higher compressibility. This substantially improved powder grade was traded under the name MH100.24 which was the forerunner of today’s NC100.24.

During World War II, iron powder metallurgy went through a dramatic development in Germany, where, due to shortage of copper, artillery shell driving bands, compacted and sintered from plain iron powder, were produced in very large quantities which in 1944 amounted to 30,000 metric tons per annum.

The iron powder for this purpose was mainly made by grinding wire cuttings and sheet clippings in hammer mills of type “Hametag”. This process yielded a powder well suited for the application, and such powder was actually continued to be produced in Czechoslovakia until 1987-89. The production costs, however, were so high that the process was very short-lived in Western Europe after the war.

In conjunction with the war effort, also other iron powder processes were developed in Germany. The most important one was the so-called RZ-process (Rohisen-Zunder-Verfahren) developed by the Mannesmann AG. Desulphurized cast-iron was atomized by air jets. The resulting powder consisted of small agglomerates of spherical particles having a heavily oxidized surface and a highly carbon-containing core. This crude powder was heated in closed iron boxes, and the oxide on the particle surface reacted with the carbon in the core, thus forming a reducing atmosphere of carbon monoxide inside the boxes. After this treatment, the powder consisted of particles having a
somewhat spongy surface and a compact core. Owing to the spongy surface of its particles, the powder had good green strength, but due its relative high amounts of residual oxides, it had poor compressibility.

**Production and use of RZ-type** iron powders have virtually ceased today. A modified version of the old RZ-process, however, was used by the Quebec Metal Powders Corporation of Canada for the production of some of their Atomet powder grades. Also here, the basis of the process is a liquid cast-iron which, differing from the old RZ-process, is atomized by a water jet instead of an air jet. The resulting crude powder was ground and mixed with iron oxide. In a subsequent annealing process under reducing conditions, carbon and oxygen are eliminated from the mix. Although this type of powder is still in production, it is gradually giving way to powder types obtained by water atomization of liquid low carbon iron.

**Immediately after the war,** Husqvarna Vapenfabriks AB of Sweden developed an iron powder process on the basis of electrolytic iron. This electrolytic iron powder, being extremely pure and having excellent compressibility, was ideally suited for the production of high-density components. However, because of its high manufacturing cost, it found only a very limited market, and its production was discontinued in the early seventies.

**Since 1950,** Höganäs sponge iron powders have also been produced by Höganäs Corporation in Riverton, N.J., USA.

**In 1958,** Esamann AB of Sweden, a manufacturer of welding electrodes, started to produce iron powder by water-atomization of liquid low-carbon iron. This powder had a high bulk density and was used as a component in the coating of the company’s welding electrodes.

**In 1962,** the A.O. Smith Corporation in the USA was first to use water-atomization of liquid low-carbon iron and low-carbon steel, followed by an annealing process, for the manufacturing of powders for sintered structural parts. These powders were an immediate success because, due to their high compressibility, they allowed the production of high-density parts of excellent quality.

**In 1968,** Höganäs AB started the production of water-atomized iron and steel powders in Sweden (taking over the Esamann-plant) and in the USA (building a new large atomizing plant at Riverton, N.J.). Refining the water-atomizing process, Höganäs soon succeeded in making water-atomized iron powder grades of superior quality, some of them matching the compressibility of electrolytic iron powder and thus replacing the latter on the market.

**Today (1996),** Höganäs AB has production facilities for iron and steel powders in Sweden, in Belgium, in the USA, and in Japan, China, and India.
2. PRODUCTION OF IRON AND STEEL POWDERS

2.3 The Höganäs Sponge Iron Process

The Höganäs sponge iron process, is essentially a chemical process in which finely divided iron ore is being reduced with coke breeze yielding a spongy mass of solid metallic iron, which can readily be comminuted to iron powder. The iron ore used at Höganäs is a magnetite slick (powdery $\text{Fe}_3\text{O}_4$) obtained by selective mining from mines in the north of Sweden and Norway. This magnetite slick, which by nature contains only very small amounts of gang and has extremely low contents of sulfur and phosphorus, is being dressed and concentrated while still at the mining location and then delivered to Höganäs in a highly pure state.

As shown in the flow-sheet at Fig. 2.2, the process of transforming the magnetite slick to iron powder proceeds as described on the next page.
2.3 THE HÖGANÄS SPONGE IRON PROCESS

1. Reduction Mix of Coke Breeze and Limestone
2. Iron Ore
3. Drying
4. Crushing
5. Screening
6. Magnetic Separation
7. Charging in Ceramic Tubes
8. Reduction in Tunnel Kilns, Approximately 1200°C
9. Discharging
10. Coarse Crushing
11. Storage in Silos
12. Crushing
13. Magnetic Separation
14. Grinding and Screening
15. Annealing in Belt Furnace, Approximately 800-900°C
16. Equalising
17. Automatic Packing
18. Iron Ore
19. Reduction Mix

Figure 2.2. Flow-sheet for the Höganäs Sponge Iron Process.
2. PRODUCTION OF IRON AND STEEL POWDERS

The process starts with two raw materials: a "reduction mix" consisting of coke breeze blended with ground limestone (1), and a pre-processed magnetite slick (2).

The magnetite slick and the reduction mix are being dried separately in two rotary ovens (3). The slightly agglomerated dried reduction mix is crushed (4) and screened (5), and the dried magnetite slick is passed through a magnetic separator (6). Then, both materials are charged by means of an automatic charging device into tube-like ceramic retorts as illustrated (7), (18), (19). These retorts have an ID of 40 cm, are 2 m long, and consist of four tube segments of silicon carbide being stacked on top of each other.

These retorts are standing, 25 each, on rail-bounded cars which are clad with a thick layer of refractory bricks. These cars are traveling slowly through a tunnel kiln of approx. 260 m length (8) within which the retorts are gradually heated to a maximum temperature of approx. 1200°C. As the temperature inside the retorts increases, the coke breeze begins to burn forming CO which, in turn, begins to reduce the magnetite to metallic iron while itself oxidizing to CO$_2$.

The so generated CO$_2$ reacts with the remaining coke breeze forming new CO, which again reduces more magnetite to metallic iron. This reaction cycle continues until all magnetite has been reduced to metallic iron and the major part of coke breeze is burned up. Parallel to the reduction cycle, the limestone in the reduction mix binds the sulfur arising from the burning coke breeze.

After completed reduction, the retorts are slowly cooled down again to approx. 250°C before leaving the kiln. Inside each retort, there is now a tube-like sponge iron cake with a porosity of about 75%, a residue of unburned coke breeze, and a sulfur-rich ash. At an automatic discharging station (9), the sponge iron tubes are pulled out off and the remaining coke breeze and ash are exhausted from the retorts. Thereafter, the retorts are ready to be charged again and go on a new trip through the tunnel kiln.

The sponge iron tubes (after having been cleaned from adhering coke breeze and ash) are in several steps crushed and comminuted to a particle size below 3 mm (10). The so obtained crude powder is intermediately ensilaged before further processing. From the intermediate silo (11), the crude powder is passed through a specially designed chain of magnetic separators (12), mills (13) and screens (14), in order to be refined to a particle size below 150 µm (<100 Tyler-mesh) and a well defined bulk density (apparent density).

Subsequently, the powder is passing a belt furnace (15) where it is soft-annealed at 800 - 1000°C in hydrogen, and its remaining contents of carbon and oxygen are reduced to a very low level. During annealing, the powder agglomerates to a very crumbly cake which is gently comminuted again in a special mill (not shown on the flow-sheet). The so treated powder has good compressibility and high green strength.
Many belt furnaces are, of course, available to take care of the huge volume of iron powder to be treated. These modern electrically heated belt furnaces are especially designed for the purpose, the belt width being 1500 mm.

The powder from several belt furnaces is collected in a special silo (16), where it is homogenized in lots of 60 or 120 tons. Each lot is carefully checked with respect to specified properties and – if okayed – packaged and stored, ready for shipment (17).

2.4 The Höganäs Water-Atomizing Process

A modern atomizing plant of Höganäs AB is located at Halmstad, a small coastal town 80 km north of Höganäs. The water-atomizing process, as practiced there, can be followed on the flow-sheet shown at Fig. 2.3, and is described in detail on the next page.
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Figure 2.3. Flow-sheet for the Höganäs Water-Atomizing Process.

Figure 2.3. Flow-sheet for the Höganäs Water-Atomizing Process.

Legend:
1. Selected scrap
2. Arc furnace
3. Liquid Steel
4. Injection
5. Atomizing
6. Dewatering
7. Drying
8. Magnetic Separation
9. Screening
10. Equalizing
11. Transportation

5. ATOMISING
A. Tundish
B. Steel Stream
C. High Pressure Water
D. Nozzle
E. Atomised Iron Powder
2.4 THE HÖGANÄS WATER-ATOMIZING PROCESS

The raw material for this process is a carefully selected iron scrap and sponge iron from the process described in the preceding paragraph. This raw material (1) is melted down in an electric arc furnace of 50 tons capacity (2) where, if desired, alloying elements can be added.

The melt is teemed slag-free through a bottom hole into a ladle (3) where it is refined with an oxygen lance (4). The ladle is then transferred to the atomizing station (5), and the liquid iron (or steel) is teemed slag-free through a bottom hole in the ladle into a specially designed tundish (A).

From there, the liquid iron (or steel) flows in a well controlled stream (B) through the center of a ring-shaped nozzle (D) where it is hit by jets of highly pressurized water (C). The stream of liquid iron (or steel) explodes into fine droplets (E). Some of these droplets freeze immediately to small spheres, others unite in small irregularly shaped agglomerates while freezing.

Air, swept along by the water jet, and water vapor arising in the atomizing process, cause superficial oxidation of the small droplets. The solidified droplets and the atomizing water are collected in a huge container, where they are settling as a mud. This powder mud is de-watered (6) and dried (7).

The dry powder is magnetically separated from slag particles (8), screened (9) and homogenized (10), and eventually transported in special containers (11) to the works at Höganas for further processing.

In the state as leaving the atomizing plant, the atomized powder particles are not only superficially oxidized but also very hard because, due to the extremely high cooling rates residing in the atomizing process, they have solidified in the martensitic state - despite their low carbon content.

The powder is, therefore, soft-annealed, and its surface oxides and residual carbon are reduced in belt furnaces of the same type as described in the preceding paragraph. Routines for homogenizing, quality checking, packaging and storing are the same as for sponge iron powders.
2.5 Alloying Methods

In order to achieve hardenable sintered ferrous materials, carbon and other suitable alloying elements, like e.g. copper, nickel, and molybdenum, have to be introduced. While carbon is normally admixed to the iron powder in the form of graphite, metallic alloying elements are commonly introduced by either of the following two methods:

Method 1 Water atomization of the liquid iron alloy, resulting in a homogeneously alloyed powder.

Method 2 Mechanically blending plain iron powder with the respective alloying elements in powder form, and letting the actual alloying process take place during sintering of the parts compacted from the powder mix.

Both methods have their advantages and disadvantages:

**Homogeneously alloyed powders**

Advantages:
- Alloying elements do not segregate when the powder is handled.
- Yield fully homogeneously alloyed sintered parts.

Disadvantages:
- Have low compressibility, because their particles are solution-hardened. (See Figs. 2.4 and 2.6).
- In order to change or correct the composition of a fully alloyed powder, if ever so little, a new melt (usually 50 tons at time) will have to be atomized.

**Powder mixes**

Advantages:
- Have higher compressibility. (See Fig. 2.6).
- No additional mixing operation is required as the powder has to be admixed with a lubricant anyway.
- The composition of a powder mix can very easily be changed or corrected by re-mixing it with additional amounts of either iron powder or alloying elements.

Disadvantages:
- Yield less homogeneously alloyed sintered parts, because the admixed alloying elements (except carbon) diffuse very slowly in solid iron. (ref. Chapter 6, Figs. 6.9 and 6.10).
- Alloying elements tend to segregate when the powder mix is transported and handled. (However, powder mixes can be made segregation-proof by means of special treatments as described in the following paragraph).
2.6 Distaloy and Starmix

In order to eliminate the segregation problem with powder mixes, Höganäs AB has developed two special patent-protected processes for the production of segregation-proof iron powder mixes. A large variety of standard and tailor-made powder mixes produced according to these processes are offered under the trade-names Distaloy and Starmix.

The Distaloy process can be described as follows: alloying elements used in the Distaloy process are mainly copper, nickel and molybdenum (but not graphite) in the form of very finely grained powders. The process starts with weighing-in a production lot of 30 tons of iron powder and alloying powders in exactly controlled proportions. This lot is mixed in a double-cone mixer. Special precautions are taken to prevent segregation of the mix when discharged from the mixer.

The so produced powder mix is heated-treated in a continuous furnace under a reducing atmosphere at a temperature somewhat below the melting point of the lowest-melting alloying element. During this heat-treatment, the fine particles of the added alloying elements are safely agglomerated to the surfaces of the coarser iron powder particles.

The diagram at Fig. 2.4 shows the influence of alloying elements on the hardness of iron.

![Diagram showing the influence of alloying elements on the hardness of iron.](image-url)
See SEM-photographs at Fig. 2.5. Due to inter-diffusion between the agglomerated alloying particles and the iron particles, the latter become, to a certain extend, locally pre-alloyed.

Figure 2.5. SEM-photographs showing fine particles of copper, nickel and molybdenum agglomerated, in the Distaloy process, to the surface of an iron powder particle.
The so treated powder mix contains the alloying additives as finely and evenly dispersed as possible and is entirely segregation-proof. The fact that the iron powder particles are locally pre-alloyed has practically no negative effect on the compressibility of the mix. See Fig. 2.6, where the compressibility curve for a Distaloy powder is shown in comparison with those for an ordinary powder mix, and for an atomized homogeneously alloyed powder of identical compositions.

Graphite and lubricants have to be excluded from the Distaloy process because, during heat-treatment of the powder mix, graphite would carbonize the iron particles and spoil the compressibility of the powder mix, and lubricants would burn-off.

Figure 2.6. Compressibility curves for three ferrous powders produced by different methods but having the same chemical composition: 1.75% Ni, 1.5% Cu, 0.5% Mo, remainder Fe.
2. PRODUCTION OF IRON AND STEEL POWDERS

The Starmix process uses special types of organic binders to glue graphite, lubricants and other additives safely to the iron powder particles during the mixing procedure. See SEM-photograph at Fig. 2.7.

Figure 2.7. SEM-photograph showing fine graphite particles glued, in the Starmix process, to the surface of an iron powder particle (NC 100.24).
The Starmix process can, of course, be applied to ordinary iron powder mixes as well as to Distaloy powders; in both cases, it yields segregation-proof press-ready powder mixes. The gain in product consistency achieved through the Starmix process is illustrated in the diagrams at Figs. 2.8 to 2.11.

Figure 2.8. Graphite loss in air de-dusting test for a Starmix-treated powder mix and for a conventional powder mix.

Figure 2.9. Relative frequency of dimensional changes during sintering for a Starmix-treated powder mix and for a conventional powder mix.
2. PRODUCTION OF IRON AND STEEL POWDERS

Figure 2.10. Variation of carbon content in sintered parts during mass production, comparing Starmix-treated and conventional mixes of NC100.24 + 1.2% graphite.

Figure 2.11. Comparison of powder properties and sintered properties for Starmix-treated and conventional powder mixes of NC100.24 + 0.8% graphite + 0.8% Zn-stearate.