KAWASAKI STEEL TECHNICAL REPORT
No.3 (September 1981)

Atomized Iron Powder Plant and Product Quality

Takafumi Fujikawa, Yoshiaki Maeda, Seiji Yasuda, Hiroshi Nonaka, Shunji Ito, Minoru Nitta

Synopsis :
In April 1978, Welding Rod and Iron Powder Plant at Chiba Works of Kawasaki Steel Corp. began manufacturing of atomized iron powder for powder metallurgy with a 400t/month capacity as the first stage. Various types of steels melted in the 5t electric furnace are pulverized into iron powder with the water atomizing method developed by Research Laboratories. Exceeding commercial-grade atomized iron powder in purity, compressibility and compatibility, this iron powder is best suited to high density parts for use in powder metallurgy. Special attention is paid to the following points in its industrial use: (1) Manufacture of iron powder of stable quality, (2) Prevention of environmental pollution caused by dust generation and water effluence, (3) Automatization and labor-saving methods centered on transportation of slurry and iron powder, and (4) Prevention of foreign powder mixing.

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In April 1978, Welding Rod and Iron Powder Plant at Chiba Works of Kawasaki Steel Corp. began manufacturing of atomized iron powder for powder metallurgy with a 400 t/month capacity as the first stage. Various types of steels melted in the 5 t electric furnace are pulverized into iron powder with the water atomizing method developed by Research Laboratories. Exceeding commercial-grade atomized iron powder in purity, compressibility and compactibility, this iron powder is best suited to high density parts for use in powder metallurgy. Special attention is paid to the following points in its industrial use:

(1) Manufacture of iron powder of stable quality,
(2) Prevention of environmental pollution caused by dust generation and water effluence,
(3) Automatization and labor-saving methods centered on transportation of slurry and iron powder, and
(4) Prevention of foreign powder mixing.

1 Foreword

The iron powder plant has since 1966 been manufacturing reduced iron powder using mill scale as the raw material. During these years, demand for iron powder has been increasing along with the development of automobiles, electrical appliances, business machines and many other types of industrial equipment. This plant has managed to meet the demand by a series of expansions and improvements in its equipment.

However, the development of powder metallurgy technology and the diversification of its application have demanded the development of powder suited for parts of higher strength and density than those of reduced iron powder used at present. To cope with this trend, we started the construction of an iron powder plant in May 1977 based on the "pencil-jet" water atomizing method which had been in the process of development by Kawasaki Steel Research Laboratories since around 1970. The first period of construction work for the atomizing plant was completed in April 1978, to be followed by the operation.

The conventional mill-scale reducing process poses difficulties in the manufacture of alloy steel powders and is unsuitable for the manufacture of the iron powders used for those parts which require higher density, because of the sponginess and irregularity of the iron powder particles obtained. The atomizing process, however, upon having added alloying materials to the target values in an electric furnace, pulverizes a teemed molten steel flow directly with high pressure water. This makes it suitable not only for the manufacture of various alloy steel powders but for the production of iron powders for high density parts owing to the small number of pores within particles.

The operation of this equipment has made the iron powder plant to be the first in Japan to manufacture both reduced and atomized iron powders which each has unique features. The production capacities are as follows:

(1) Atomized iron powder
For powder metallurgy: First period: 400 t/month
(when completed, about 1 200 t/month)
(2) Mill-scale-reduced iron powder
For powder metallurgy: 1 700 t/month
For welding rod and others: 200 t/month

Besides pure-iron powder, the atomized iron powder includes alloy steel powders such as 3Cr steel powder (Cr-Mo-V steel), 6.5Co steel powder (Ni-Mo-Co steel), 4 100 steel powder (Mn-Cr-Mo steel), and 4 600 steel powder (Ni-Mo-Cu steel).

The present finish reduction process produces iron

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** Welding Rod & Iron Powder Plant
*** Research Laboratories

powder for use in the powder metallurgy by stationary reduction using AX gas. However, Kawasaki Steel is planning to construct in the future a plant based on VIDOP (Vacuum Induction-heating De-Oxidation Process) capable of manufacturing a low-oxygen alloy steel powder with excellent hardenability and toughness. The process is of Kawasaki's own development and the plant will supply a new steel powder as the raw material for high-strength parts.

The outline of the equipment and the product quality obtained are given below.

2 Equipment

2.1 Manufacturing Process

The outline of the manufacturing process is shown in **Fig. 1**. Molten steel which has been prepared in a 5 t electric furnace is transported into the teeming yard, where the teeming operation is conducted while keeping the temperature inside the tundish constant by the use of a ladle heating furnace, and simultaneously atomizing is carried out with a high-pressure water. The iron powder slurry produced in the atomizing chamber is sent continuously to the settling tank by a slurry pump, to be separated into water and thick iron powder slurry. After having been primarily dehydrated by a vibration dehydrator, the thick iron powder slurry is dried with a steam dryer. After forced cooling to room temperature by a cooler, it is crushed, magnetically separated and classified to become as-atomized powder. This as-atomized powder undergoes a finish reduction with AX gas in the finish reduction furnace as in the case of the reduced iron powder, and is then crushed, classified, and sieved to become an atomized iron powder.

2.2 Outline of the Equipment

2.2.1 Electric furnace

Photo. 1 shows an electric furnace in the process of tapping. Scrap and alloying materials etc., are melted and so refined that the molten steel will have aimed composition and temperature. The furnace is an arc furnace for steel making with a nominal capacity of 5 t and the rating of the transformer is 3 000 kVA.

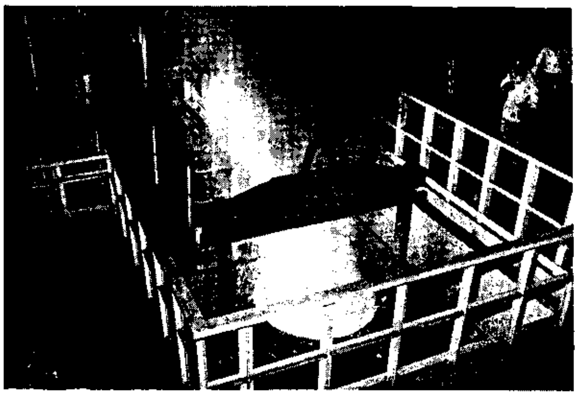


Photo. 1 Tapping at 5 t electric furnace

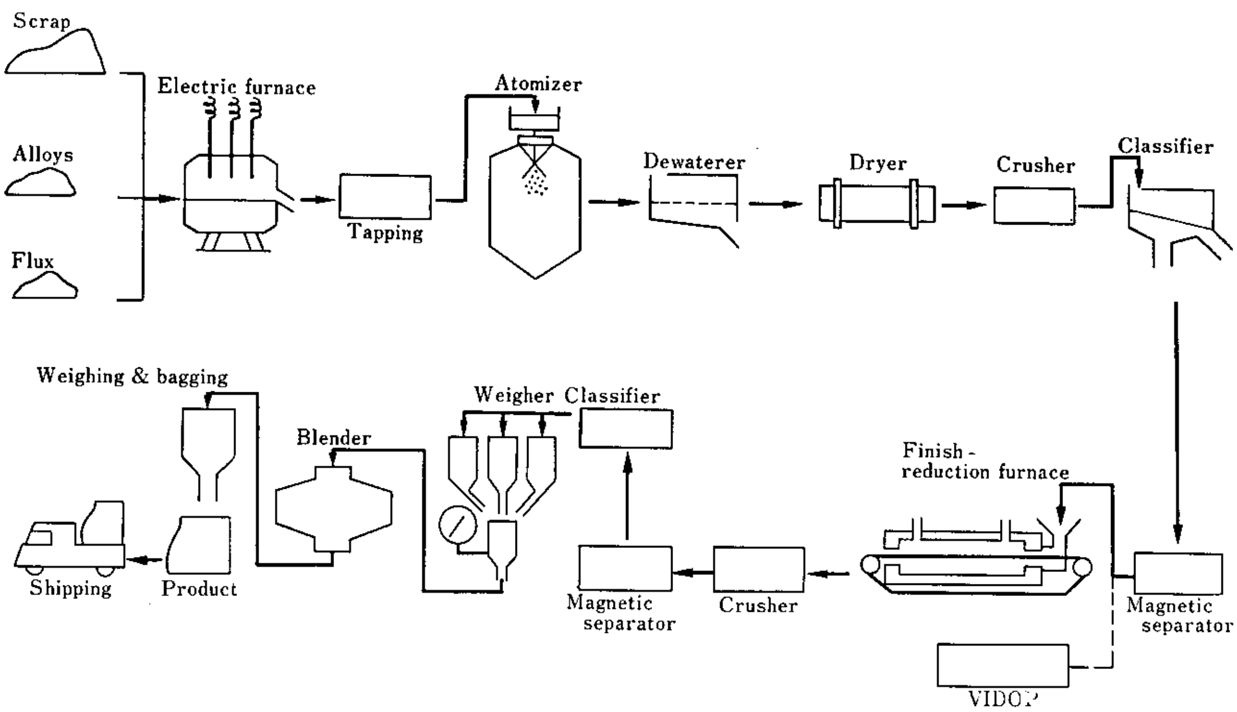


Fig. 1 Manufacturing of atomized steel powder

2.2.2 Ladle heating furnace

In this equipment, the molten steel temperature in a tundish during teeming is adjusted to aimed value by heating the molten steel in a ladle. This is aimed at stabilizing the iron powder quality and reducing the nozzle clogging. The transformer has an 800 kVA rated capacity and permits a temperature increase of 2°C/min.

2.2.3 Teeming equipment

This equipment has a large effect on particle size and oxygen content of iron powder. Since the molten steel and water are brought into contact with each other during operation, some measures were required for stable quality and safety precaution to prevent steam explosion.

Fig. 2 gives an assembly drawing of the overall teeming equipment.

(1) Atomizing device
Fig. 3 indicates an assembly drawing. High-pressure water nozzles are arranged in a circle at the top part of the atomizing chamber. Atomization of the molten steel is attained by dropping the molten steel exactly on the focus of the conical film. To prevent oxidation of the molten steel during atomization, the entire chamber is designed to be airtight and obtain a complete displacement by inert gas. This is also provided with draft pipe and pressure equalizing pipes to eliminate a pressure fluctuation in the vicinity of the high-pressure water film and a pressure difference between upper and lower parts, in order to prevent the disorder of the high-pressure water film which would affect the particle size of the iron powder product.

(2) Oxidation preventing device
The H_2 loss value of as-atomized powder which affects the product quality and the productivity of the finish reduction furnace is virtually determined by the oxygen concentration in the atmosphere under which the molten steel is atomized. In order to lower the oxygen concentration it is necessary to make the equipment airtight to prevent air inflow through a gap between the bottom surface of the tundish and the top of the chamber. A method is employed whereby air is mechanically shut off to lower the oxygen concentration in the chamber during teeming so that a product with a low H_2 loss can be obtained.

(3) Nozzle centering device
This is a device to allow molten steel flow from the tundish to fall exactly on the focus of the high-pressure water film. A center mark is provided in the preparation platform located off the teeming position. Positioning is made on the preparation platform after the tundish is set on the tundish car, so that molten steel flow during the teeming operation will fall exactly on the focus of the high-pressure water film. Two tundishes are always provided, one of which standing by for emergency.

(4) Molten steel flow control device
The teeming flow rate of molten steel varies with the tundish nozzle diameter and the depth of the molten steel in the tundish. Since the depth is usually observed and adjusted by a worker, it is

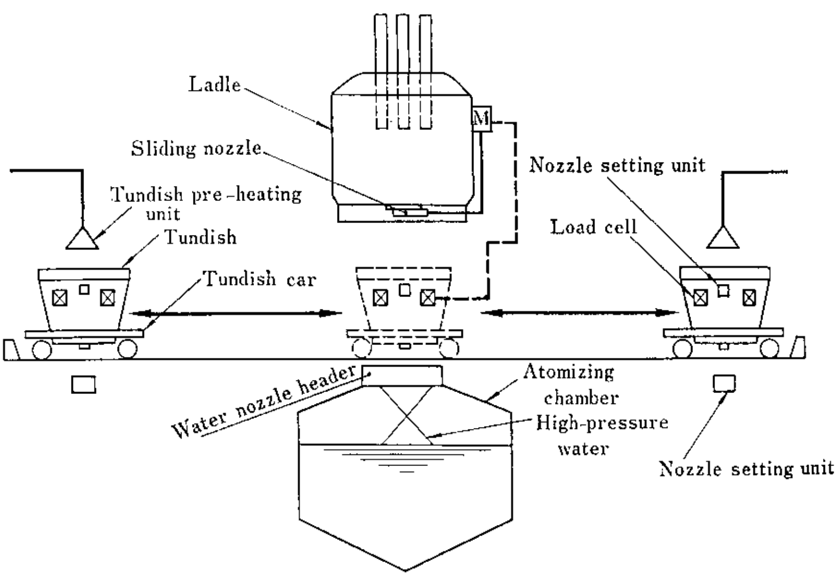


Fig. 2 Equipment for pouring and atomization

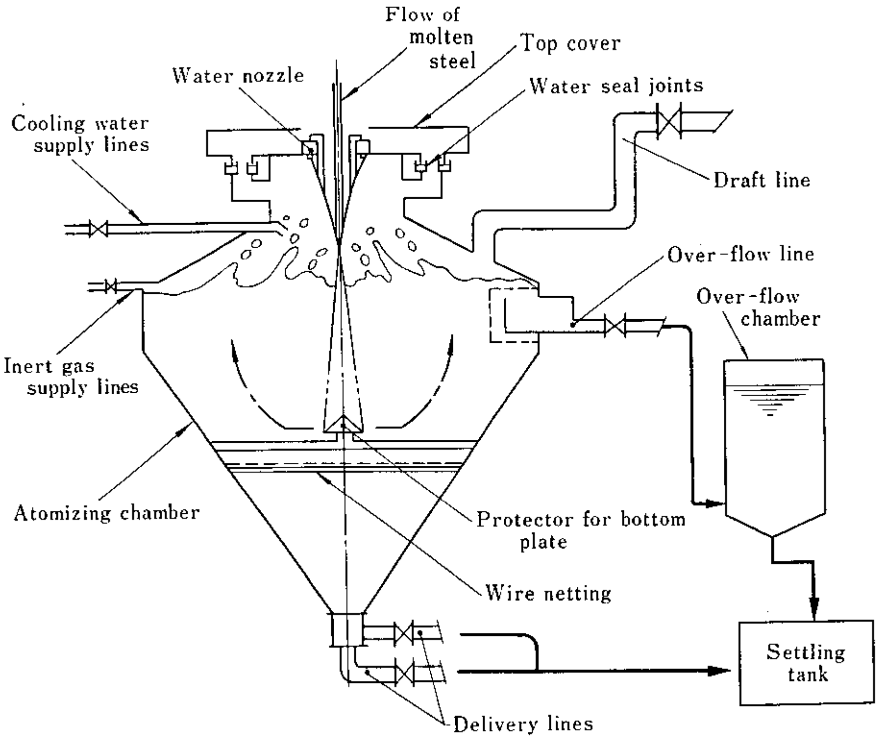


Fig. 3 Schematic drawing of atomizing chamber

bound to have variations from worker to worker. This device is automatically to measure the quantity of the molten steel in the tundish and change the flow rate of the molten steel from the ladle in order to eliminate these variations due to manual handling and obtain iron powder with uniform particle size.

Detecting device	:	4-point load cell
Detectable weight	:	± 10 kg
Precision	:	within ± 10 mm from standard level

(5) Safety device

(a) Molten steel handling

The sliding nozzle of the ladle is opened and closed by an electrical drive; an air motor is also provided to cope with possible power failures. The molten steel in the tundish can be discharged into an off-line emergency trough by running the tundish car or using the auxiliary trough. If the sliding nozzle should fail to close, discharge of the molten steel in the ladle will be conducted in the same manner.

(b) Control of water quantity and temperature

Increase in the water temperature causes unfavorable change of the molten steel flow due to pressure changes in the chamber, while a drop of the water level causes impairment of the bottom of the atomizing chamber, resulting in accidents. For this reason, the water temperature and the water level are measured during operation and, according to their changes, water supply is automatically adjusted. In case of emergencies such as power failure, the overhead water tank will supply water until molten steel handling has been finished.

2.2.4 Dehydration and drying devices

(1) Vibration dehydrator

Slurry discharged from the setting tank contains approximately 35 wt% water and if it is thrown directly into the dryer, it will stick to the iron slurry transfer machine and the inside of the dryer, causing problems of undesirable equipment maintenance and foreign steel powder inclusion. Solution of these problems requires the installation of a dehydrator to reduce the water content to 10% or under. The dehydrator installed here was originally developed by Kawasaki Steel as the most suitable dehydrating machine for iron powder.

It has the following features:

(a) The method in which iron powder is exposed to the air during dehydration causes the problem of oxidation. On the other hand, this device performs dehydration below the water surface, producing little oxidation.

(b) Since this device is of a simple box form, it can be washed easily leaving no residue.
Principal specifications are as follows.

Volume	: 4.4 m^3 (box type)
Capacity	: 6.5 t/40 min
Vibration force	: 30.0 t
Frequency	: 1 450 cpm
Amplitude	: 1.7–2.0 mm
Dehydration efficiency	: Water content before dehydration 35 wt% Water content after dehydration 10 wt% max

(2) Steam dryer
This device dries dehydrated iron powder containing water of 10 wt% max. Since it has a good thermal efficiency and drying progresses at a temperature of not more than 200°C, it permits little oxidation. It is also a batch-type steam tube dryer which can operate in an atmosphere of inert gas during drying. The inert gas can be recycled for economy sake. The oxygen concentration during drying can be controlled at not more than 1%. Principal specifications are shown below:

Type : Steam tube dryer 2 440 mm ϕ $\times$ 4 500 mm
Steam: 7 atg
Operation: Batch type
Water content attained: 0.02% max
Scrubber: Capacity of 600 m^3/h
Preheater: Steam heater

2.2.5 Classifying equipment

The process diagram of the classifying equipment is shown in **Fig. 4**, and the appearance of the equipment in **Photo. 2**. The main purpose of this equipment

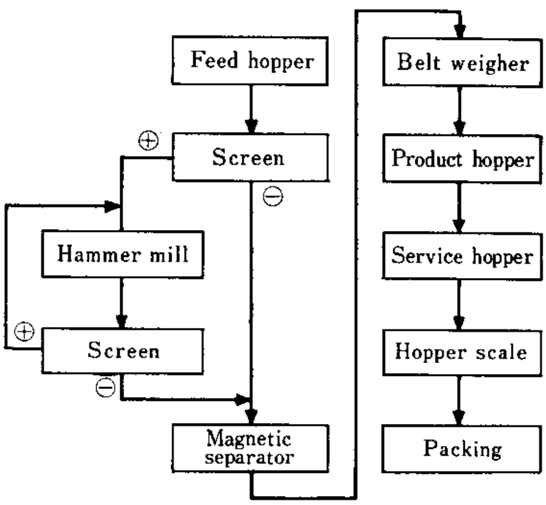


Fig. 4 Process diagram of the crushing and sieving system

Photo. 2 General view of crushing and sieving facility

is the crushing of agglomerated particles which have been produced during atomizing, the classification for particle size stabilization, and the magnetic separation for removing slag and other inclusions. The quantity recycled to the hammer mill is controlled to ensure adequate crushing. A vibration conveyor and a special distributor are employed to eliminate the need for belt conveyors and gate portions in the piping, thereby reducing foreign steel powder inclusions and adhesion of iron powder to the machine body.

Main specifications:

Equipment capacity: 3.5 t/h
Screen: 4 ft $\times$ 10 ft $\times$ 2 sets
Single floor open type with cover
Hammer mill: 500 mm ϕ $\times$ 300 mm
30 kW $\times$ 4 p
Magnetic separator: Magnetic separation
1 300G max
Feeding weigher: Precision $\pm 1/100$
Hopper scale: Precision $\pm 1/500$, load cell type

2.2.6 Drainage treating device

The water recirculating diagram is shown in **Fig. 5**, and the appearance of the equipment in **Photo. 3**.

The drainage includes the following two kinds: atomizing water which contains iron powder and the cooling water for general equipment and devices. The latter is water with an added rust inhibitor, small in

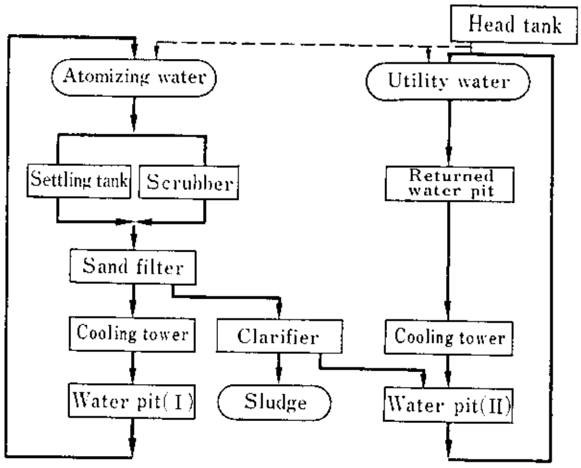


Fig. 5 Process diagram of recirculating water

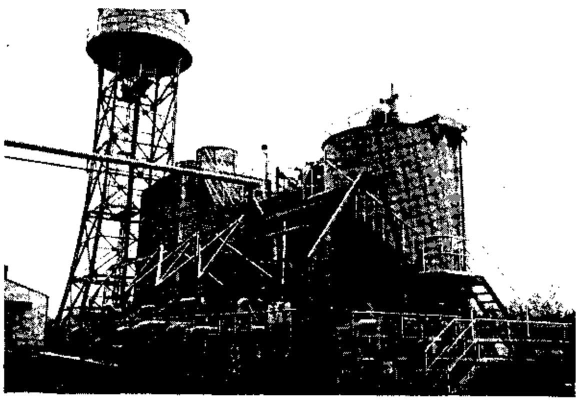


Photo. 3 General view of recirculating water facility

content of suspended solid, and can be readily recirculated, whereas the atomizing water must be filtered to reduce the content of suspended solid consisting of iron powder to 15 ppm or under for prevention of wear to the impeller of the high-pressure-water pump and the atomizing nozzle. The sludge water which is formed during washing in the filter tower is also treated in the thickener, the supernatant liquid being used as a makeup water for equipment and device cooling water system, and the sludge being recovered as an iron source in the sludge plant, so that no drainage is discharged outside the works.
Main specifications:

(1) Water treating system No. I (for atomizing water)
  Recirculation rate: 180 m^3/h
  Sand filter tower : Pressure type 40 $m^3/h \times 2$ sets, 1 900 mm $\phi \times$ 5 000 mm, SS < 15 ppm
  Cooling tower : Inducing and forced draft type, wooden frame, slate roofed, rectangular type
  Clarifier : 4 800 mm $\phi \times$ 5 320 mm, SS < 15 ppm
  Recirculating water tank: 365 m^3

(2) Water treating system No. II (for equipment and device cooling water)
  Recirculation rate: 120 m^3/h
  Cooling tower : Inducing and forced draft type, wooden frame, slate roofed, rectangular type
  Recirculating water tank: 92 m^3
  Returned water tank : 34 m^3

2.2.7 Accessory equipment

(1) Electric furnace dust collector
There are several suction positions such as for local hoods around the furnace, building hoods and ladle heating furnace hoods. The necessary suction rate varies depending on the time of refining and the multiplicity of operations conducted at a time. A new dust collector which combined a two-pole (8 p–6 p) conversion system in the exhaust blower motor and a damper opening control was developed for saving energy and installed.
  Type: Pressure-type bag filter
Exhaust blower
  Capacity : 3 900 m^3/min × 410 mmAq at 80°C
  Electric motor: 500/210 kW 985/740 rpm
  Dust collecting chamber: 6 chambers, filtration area: 3 880 m^2

(2) Classifying equipment dust collecting device
  Type: Suction-type bag filter
Exhaust blower
  Capacity : 400 m^3/min × 300 mmAq at 40°C
  Electric motor: 37 kW, 2 030 rpm
  Dust collecting chamber: 12 chambers, filtration area: 324 m^2

2.2.8 Finish reduction equipment

As-atomized powder undergoes such treatments as reduction annealing and sieving by means of the finish reduction furnace and related accessory equipment to become atomized iron powder.

The purpose of the finish reduction is to remove such impurities as C, N, S, and O in as-atomized powder and eliminate strain due to quenching in the atomizing in order to obtain powder suitable for powder metallurgy.

Some alloy steel powders contain a larger amount of those components which have stronger affinity with O than Fe (for example, Mn, Cr, etc.). Therefore in the finish treatment for those products, it is necessary to give a high temperature and sufficiently lower the dew point of the atmospheric gas inside the furnace in order to prevent oxidation of the as-atomized powder. For this purpose, the furnace temperature is set at about 1 000°C, and a dryer is provided to lower the dew point of the input atmospheric gas to 0°C or less.

Since atomized pure iron powder, alloy steel powder and mill scale-reduced iron powder are treated in the same line comprising the finish reduction furnace, sieving, mixing, and packing devices, these devices are designed with adequate consideration in preventing of mutual mixing.

(1) Finish reduction furnace
- Type : Radiant tube heating, steel belt continuous type
- Furnace dimension: Furnace length 49 m, belt width 1 250 mm
- Temperature : 1 000°C max
- Reducing gas: Ammonia cracking gas

(2) Accessory equipment
- Crushing machine: Hammer type 2 t/h
- Magnetic separating machine: Drum rotating type 2 t/h
- Classifying device : Vibration type 2 t/h
- Mixing machine : Double cone type 10 t/batch
- Packaging machine: Automatic weighing type 25–1 000 kg/bag

3 Quality

The features of atomized iron powder (**KIP** 300A) for powder metallurgy will be given here. As for alloy steel powder, its properties will be described only up to the stage of its compressed powder.

3.1 Chemical Composition

Kawasaki Steel atomized iron powder (**KIP** 300A) has a very high purity compared with conventional atomized iron powders and Kawasaki Steel's mill-scale-reduced iron powder (**KIP** 270MS) which is of the highest purity among reduced iron powders on the market. This is because, for the manufacture of **KIP** 300A, relatively a better quality scrap among those produced in the Chiba Works is selected and then refining is also carried out in an electric furnace aiming to reduce impure elements. **Table 1** gives examples of compositions of the above-mentioned three kinds of the pure iron based powders and the atomized alloyed steel powders marketed to date by Kawasaki Steel. For reference, one example of composition is given for the low-oxygen atomized alloy steel powder produced by the VIDOP equipment now under development by Kawasaki Steel Research Laboratories.

3.2 Properties of Powder

Photo. 4 gives the appearance of the particle as viewed by the scanning electron microscope. **Photo. 5** gives the cross-section view of the particles of both an atomized iron powder and a mill-scale-reduced iron powder. **Table 2** gives examples of apparent density, fluidity and particle size distribution of those products.

Atomized iron powders are higher in apparent density and better in fluidity than mill-scale-reduced iron powders because atomized iron powders are denser owing to fewer pores in their particles and smoother in their particle surfaces. As for particle size distribution, they are richer in both coarse particles and fine particles than mill-scale-reduced iron powders, thus showing

Table 1 Chemical composition, hydrogen loss and acid insoluble of iron powders

		Chemical composition (%)												H_2 loss (%)	Acid insolubles (%)
		C	Si	Mn	P	S	Cu	Ni	Cr	Mo	Co	V	O		
Pure iron	**KIP** 300A	0.004	0.01	0.10	0.005	0.007	—	—	—	—	—	—	—	0.10	0.06
	Commercial*	0.004	0.01	0.20	0.011	0.011	—	—	—	—	—	—	—	0.20	0.09
	KIP 270MS**	0.004	0.02	0.25	0.006	0.006	—	—	—	—	—	—	—	0.12	0.18
Alloyed steel	**KIP** 4600	0.010	0.02	0.13	0.004	0.008	0.54	1.52	—	0.50	—	—	0.16	—	—
	KIP 4100	0.04	0.03	0.76	0.007	0.005	—	—	0.97	0.23	—	—	0.55	—	—
	KIP 4100V***	0.12	0.06	0.82	0.019	0.007	—	—	1.06	0.25	—	—	0.088	—	—
	KIP 30CRA	0.05	0.03	0.13	0.007	0.005	—	—	2.98	0.28	—	0.31	0.75	—	—
	KIP 65COA	0.007	0.02	0.07	0.005	0.006	0.20	1.70	—	1.66	6.53	—	0.13	—	—

* Water atomized iron powders
** Mill scale reduced iron powders
*** Water atomized steel powders reduced by VIDOP

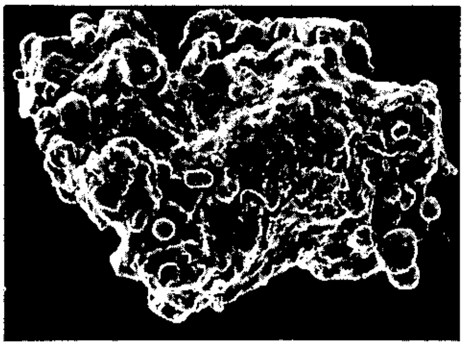
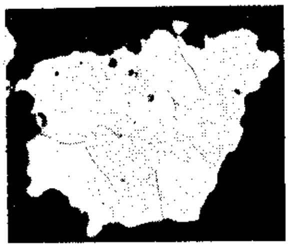


Photo. 4 Particle surface by scanning electron microscope



(a) Atomized

(b) Mill-scale reduced

Photo. 5 Cross section of iron powder particles

a wider particle distribution. The larger amount of coarser particles in atomized iron powders is due to the fact that atomized iron powder is sieved into an aimed particle size of 80 mesh max, whereas mill-scale-reduced iron powder into 100 mesh max.

3.3 Compressibility

Fig. 6 gives compressibilities of various iron powders. High density and high strength parts require an iron powder of high compressibility.

The pure-iron based atomized iron powder **KIP** 300A developed by Kawasaki Steel has a higher compressibility than in **KIP** 270MS which has the highest compressibility among reduced iron powders; it shows a compressibility approximately equivalent to or higher than other atomized iron powders now on the market. This is due to the fact that the **KIP** 300A gives softer powder due to its higher purity and consists of less porous particles.

3.4 Compactibility

In the manufacturing process of sintered parts, fracture, tipping and crack of green compact are problems which may occur, in particular, in the compacting of parts of a complicated shape or those with thin-walled portions and in the transport of green compacts to the next sintering process. Rattler value and green strength are indices representing the degree of these defects. The Rattler value represents the stability in a sharp portion of green compact; smaller values give better compactibility. The green strength represents the strength of green compact; higher values give better compactibility.

Table 2 Particle size distribution, apparent density and flow rate of iron powders

		Particle size distribution, mesh (%)							A.D. (g/cm^3)	F.R. (s/50g)
		mesh +80	−80/+100	−100/+150	−150/+200	−200/+250	+250/+325	−325		
Pure iron	**KIP** 300A	0.1	6.3	21.8	24.8	8.1	16.3	22.6	2.92	26.2
	Commercial*	0.3	7.8	12.8	22.1	8.9	17.0	31.1	3.01	23.0
	KIP 270MS**	—	0.4	19.6	35.3	14.6	12.7	17.4	2.74	25.3
Alloyed steel	**KIP** 4600	0.1	8.6	20.5	23.5	7.9	15.4	24.0	3.05	25.0
	KIP 4100	0.1	6.3	21.6	25.3	10.1	13.2	23.4	3.20	23.6
	KIP 4100V***	3.4	15.1	25.8	24.8	7.1	12.1	11.7	3.23	23.0
	KIP 30CRA	Tr	2.8	12.0	20.0	8.6	18.4	38.2	3.14	23.6
	KIP 65COA	0.1	5.0	20.9	23.3	11.4	14.3	25.0	2.84	25.0

* Water atomized iron powders
** Mill scale-reduced iron powders
*** Water atomized steel powders reduced by VIDOP

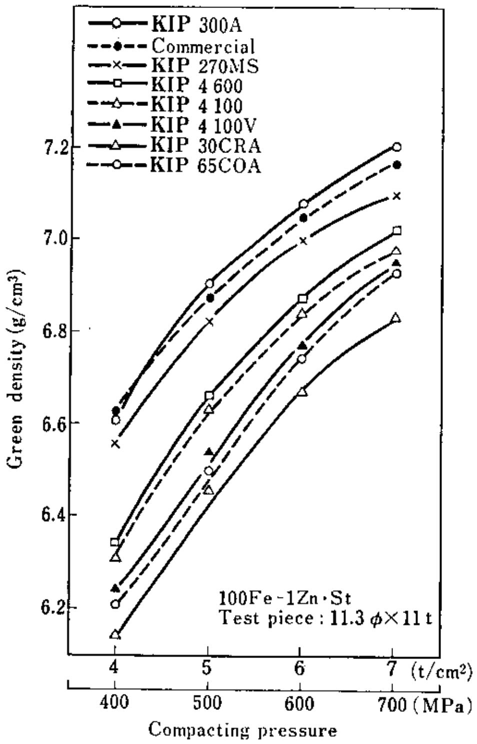


Fig. 6 Compressibility of iron powders

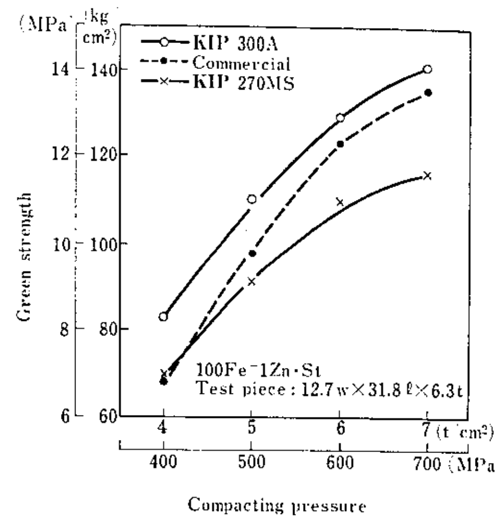


Fig. 8 Green strength of compact

Fig. 7 gives Rattler values; **Fig. 8** shows green strengths of only the pure-iron based powders (according to ASTM B312-64, about 3 900 psi at a compacting pressure of 30 tsi). **KIP** 300A has a significantly improved value in both Rattler value and green strength, compared with **KIP** 270MS and other manufacturer's atomized powder.

3.5 Sintering Properties

At the plants of customers, the iron powder is added to and blended with various alloying elements and the mixture is charged into a die. After having been compacted, it is sintered to make a sintered part. General sintering properties of the pure-iron based atomized iron powder, **KIP** 300A, will be described here. The measuring conditions for sintering properties are as follows:

Composition	:	96.1 Fe-3Cu-0.9C + 0.75 Zn·St
Sintering atmosphere	:	RX gas (butane-denatured gas D.P. 4°C)
Sintering temperature and time	:	1 130°C for 25 min

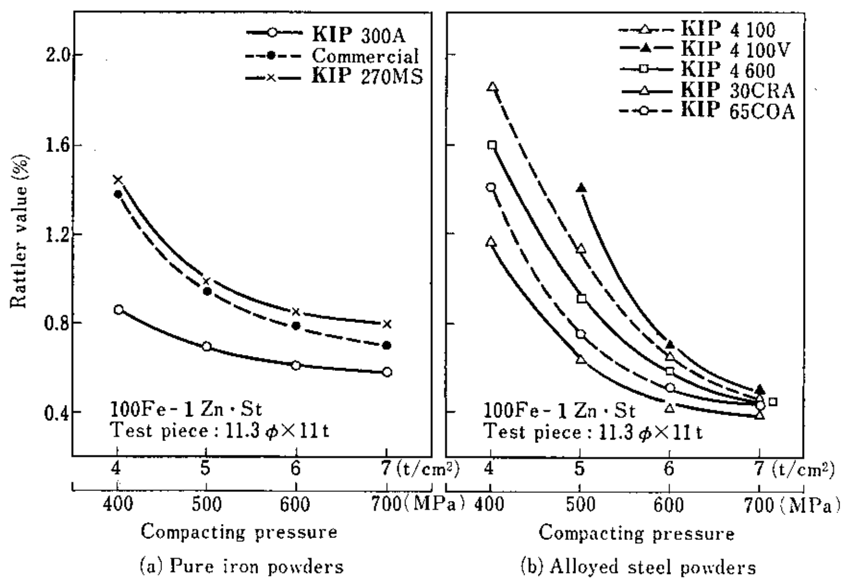


Fig. 7 Rattler value

3.5.1 Dimensional change

Fig. 9 gives the aspect of dimensional changes. Dimensional change depends on the blending ratio of addition elements and the sintering cycle. However, under the above-mentioned conditions, the changes show the same trend both in the outside and inside diameters; **KIP** 300A shows intermediate dimensional changes between those of **KIP** 270MS and commercially available atomized iron powders.

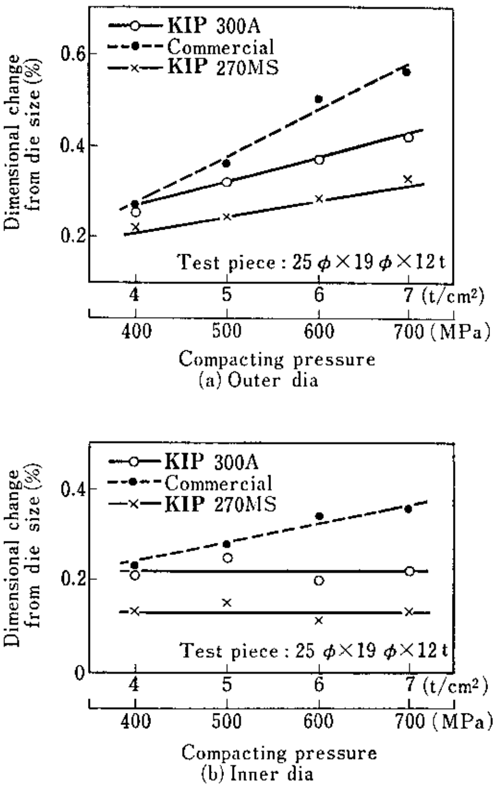


Fig. 9 Dimensional change of compact by sintering

3.5.2 Mechanical strength

Fig. 10 shows sinter density, **Fig. 11**, sinter hardness, **Fig. 12**, tensile strength of sinter, and **Fig. 13**, elongation of sinter. The maximum value of sinter density is given by **KIP** 300A. As to the strength of sintered compact in terms of hardness, tensile strength and elongation, **KIP** 300A shows values equivalent to those of other atomized iron powders commercially available and **KIP** 270MS.

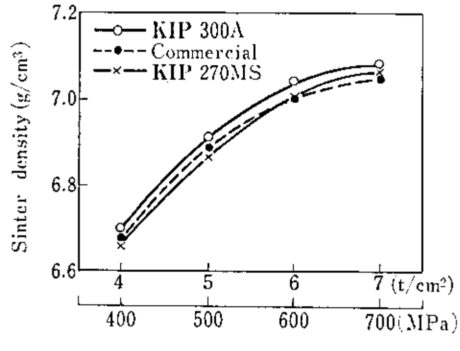


Fig. 10 Sinter density

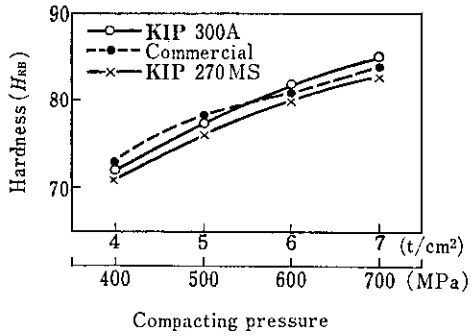


Fig. 11 Sinter hardness

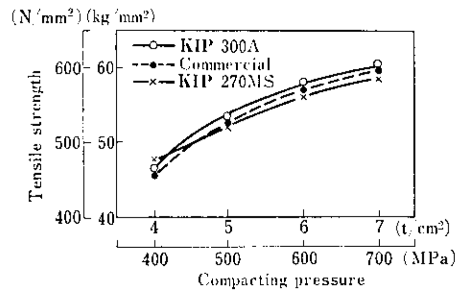


Fig. 12 Tensile strength of sinter

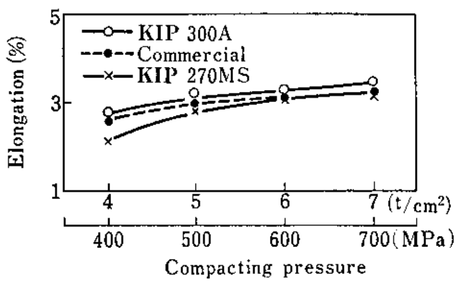


Fig. 13 Elongation of sinter

4 Conclusion

This atomizing plant and a series of related processes are the result of Kawasaki Steel's industrialization of its up-to-date total technology based on fundamental data and knowledge obtained from experiments and studies. The completion of this plant has ranked Kawasaki Steel as the only one in Japan which can manufacture both mill-scale-reduced and atomized iron powders. With three years passed from the start of its operation, this plant is showing satisfactory results steadily, and the high-quality products obtained from the stable operation exceeds the conventional atomized iron powder and mill-scale-reduced powder commercially available in terms of purity, compressibility and compactibility. Finally, the authors would like to express their hearty gratitude to those in various departments and sections concerned for their warm cooperation in the construction and operation of the subject plant.