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METHOD OF ANNEALING METAL POWDER

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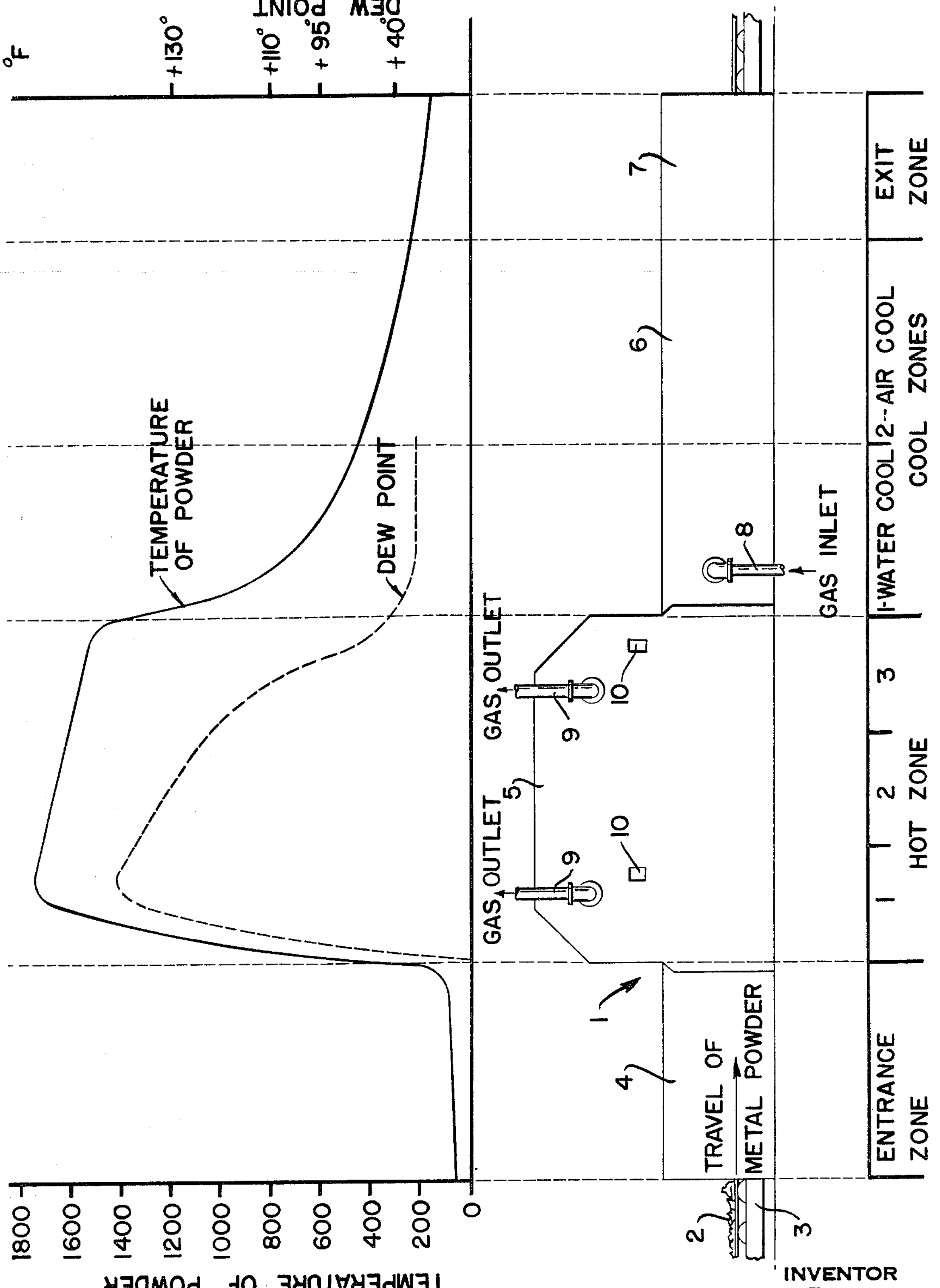

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METHOD OF ANNEALING METAL POWDER

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Int. Cl. B22f *1/00*
U.S. Cl. 148—126 12 Claims

ABSTRACT OF THE DISCLOSURE

The invention relates to a process for annealing steel powder. The powder is continuously passed through an annealing furnace and heated to a temperature in the range of 1450° F. to 2100° F. while exposed to a reducing gas. The dew point of the furnace atmosphere is maintained at a value slightly below the equilibrium value throughout the length of the heating zone by adjustment of the rate of flow of the reducing gas to the furnace.

By maintaining the dew point of the furnace atmosphere within precise limits, a substantially complete reduction of the carbon content of the steel particles is obtained and welding of the particles is prevented, so that the sintered cake of particles can be readily broken up after annealing to restore the as-atomized particle size and thereby provide an increased density for the compacted and sintered part.

This application is a continuation-in-part of application Ser. No. 671,290 filed Sept. 28, 1967, now abandoned and entitled Method of Annealing Metal Powder.

Metal powder to be used in powder metallurgy processes can be prepared by a number of different methods. Patent No. 3,325,277 of Robert A. Huseby describes a method of making metal powder having high density, as well as an irregular shape which provides substantial improvements in the physical property of the compacted and sintered part. According to the process of Patent 3,325,277, molten steel is fed by gravity in the form of a downwardly moving stream and a series of flat sheets of water are impinged against the stream of molten steel to thereby atomize the steel and provide a plurality of clump-like agglomerates of spheroidal particles. Subsequently, the particles are heated to a temperature of about 1450° F. to 2100° F. in a reducing atmosphere for a period of time sufficient to soften the particles and reduce the carbon content to a value less than 0.05%. After annealing, the sintered cake is broken up by hammermilling to restore the as-atomized particle size of the powder.

The present invention is an improvement to the process described in Patent 3,325,277 and is based on the discovery that the density of the resulting powder can be further improved by controlling the furnace atmosphere during the annealing process within precise limits.

According to the invention, the low carbon atomized steel particles are continuously fed through an annealing furnace and heated to a temperature in the range of 1450° F. to 2100° F. A reducing gas, such as dissociated ammonia or hydrogen, is supplied to the furnace and moves countercurrently to the flow of the steel powder. The dew point of the furnace atmosphere is maintained at a value slightly below the equilibrium value throughout the length of the heating zone by adjustment of the flow of the gas into the furnace. By proper control of the dew point, softening of the steel powder and reduction of the carbon content of the steel powder is obtained with a minimum flow of reducing gas. Moreover, the regula-

tion of the dew point prevents welding of the particles so that the particles can be readily broken up to restore the as-atomized particle size after annealing and thus, the density of the resulting powder is correspondingly increased.

Other objects and advantages will appear in the course of the following description.

The drawing is a schematic representation of an annealing furnace used for the invention and including curves indicating the temperature of the powder and dew point of the furnace atmosphere throughout the length of the furnace.

There are a number of basic processes by which metal powder to be used in metal processes can be prepared. For example, metal powder can be prepared by an electrolytic process, an ore reduction process, a gas atomization process or water atomization processes. Patent 3,325,277 of Robert A. Huseby describes an improved water atomization process for forming steel particles having a high density as well as an irregular shape which provides a substantial improvement in the physical properties of the compacted and sintered part. The steel to be atomized contains less than 3.3% of total alloying elements and has a carbon content of less than 0.2% and generally in the range of 0.06% to 0.10% by weight. The alloying elements can be manganese, nickel, cobalt, chromium, molybdenum, tungsten, and the like.

According to the process of Patent 3,325,277, the steel is contained within a tundish at a temperature of about 3100° F. and flows by gravity through an outlet slot or nozzle. Water is directed against the stream of molten metal at an angle of about 15° to 55° from the vertical. The water which is in the form of flat sheets or curtains serves to atomize the molten steel to produce chain-like or clump-like agglomerates of generally spheroidal particles. The steel powder, as atomized, has a particle size such that at least 85% will pass through an 80 mesh sieve and at least 75% will pass through a 100 mesh sieve.

Following the atomization, the steel is subjected to an annealing treatment which serves to soften the particles, reduce the oxide film and reduce the carbon content. To provide this annealing treatment, the powder is continuously passed through an annealing furnace and heated to a temperature in the range of 1450° to 2100° F. and preferably from 1650° to 1800° F. in a reducing atmosphere consisting of dissociated ammonia or hydrogen. The hydrogen content of the reducing gas supplied to the furnace should comprise at least 95% by volume of the active gaseous constituents, with the term active constituents excluding any inert constituents such as nitrogen. Appreciable amounts of carbon monoxide or carbon dioxide should be avoided. Carbon dioxide is an oxidizing gas, and the addition of carbon dioxide to the furnace atmosphere will tend to increase the residual oxygen content of the powder above the desired limits, thereby adversely affecting the properties of the sintered powder. The addition of carbon monoxide to the furnace atmosphere may cause carburizing of the steel.

The reducing gas supplied to the furnace is substantially completely dry, having a dew point below −40° F., and generally about −50° F.

The annealing treatment serves to soften the steel particles as well as reduce the carbon content to a value below 0.05% and generally to a value in the range of 0.001% to 0.02%. To obtain optimum ductility and subsequently obtain the maximum density for a given compaction pressure and increased physical properties in the sintered product, the powder is normally held at the annealing temperature for a period of at least one hour and generally in the range of one to two hours.

It has been discovered that by use of the above-described furnace atmosphere and by controlling the dew point of the furnace atmosphere within precise limits, complete reduction of the carbon content can be obtained without welding of the particles together, so that after annealing, the sintered cake can be readily restored to its as-atomized particle size by hammermilling.

It is believed that at the annealing temperature the following reversible reaction occurs:

$$FeO+H_2 \leftrightharpoons 2Fe+H_2O$$

In this reaction, hydrogen of the dry reducing gas reacts with the iron oxide to form water and iron. As the reduction continues and water is formed, the moisture content or dew point of the atmosphere increases, and if the moisture content becomes too great, the reaction is reversed and oxidation will occur. The reduction reaction can be maintained in a number of ways, as for example by increasing the temperature of the particles, increasing the gas flow, or reducing the amount or tonnage of metal power flowing through the furnace. As the control of the gas flow rate can be readily accomplished, this method is preferred in maintaining the desired moisture or dew point in the furnace atmosphere and thereby maintaining a reduction reaction.

For a given temperature, there is an equilibrium $H_2O:H_2$ ratio, or dew point, at which the above reaction is in equilibrium, and it is important to maintain the H_2O concentration slightly below the equilibrium $H_2O:H_2$ ratio for the particular furnace temperature, so that the reaction will be a reduction and oxidation will not occur. If the flow of reducing gas is too low in volume, the moisture content in the furnace atmosphere will increase so that the dew point, or $H_2O:H_2$ ratio, will be above the equilibrium value, and the reaction will then reverse and oxidation will occur. Alternate reduction and oxidation of the particles will serve to weld the particles together so that it is important that the dew point be adjusted so that a reduction reaction is maintained without alternately reducing and oxidizing of the particles.

Conversely, if the dew point or the $H_2O:H_2$ ratio is maintained considerably below the equilibrium value, a rapid reduction will occur and the particles will be completely reduced before they reach the end of the heating zone. This is not desirable, for in addition to wasting the reducing gas, it has been found that a rapid reduction will not permit additional hydrogen to penetrate into the steel particles so that the final reduction of the particles is not as complete as when the dew point is maintained just slightly below the equilibrium value. Furthermore, during the reduction, steam is discharged from the steel particles, and it is believed that the steam tends to keep the particles from welding or sintering together. However, if the reduction is too rapid and completed a substantial distance before the end of the heating zone, the discharge of steam will be correspondingly terminated and the particles will tend to weld together at the high temperatures involved. Therefore, it is important to maintain the dew point from 5° to 100° F. below the equilibrium value throughout the entire length of the heating zone so as to obtain reduction of the particles without alternate oxidation. It is preferred to maintain the dew point at a value of about 5° F. to 40° F. below the equilibrium value at the beginning portion of the heating zone and to maintain the dew point at a value of about 60° F. to 100° F. below the equilibrium value at the end of the heating zone.

In accordance with the invention, the steel powder is heated to a temperature in the range of 1450° to 2100° F. and preferably the powder is heated to a temperature of 1600° F. to 1800° F. at the start of the heating zone and the temperature is progressively reduced to a value of about 1500° F. to 1600° F. at the end of the heating zone. In combination with these temperatures, the dew point of the furnace atmosphere is maintained at a value of about 100° F. to 145° F., and preferably 130° F. to 140° F. at the beginning of the heating zone with the powder at temperature, and is progressively reduced to a value of below 80° F., and preferably 60° F. to 80° F. at the end of the heating zone.

FIG. 1 is a schematic representation of an annealing furnace 1 and having curves transposed above the furnace showing the temperatures of the particles and the dew point of the furnace atmosphere throughout the length of the furnace.

The steel powder 2 to be annealed is passed through the furnace 1 on a conventional conveyor 3. The furnace 1 includes an entrance zone 4 which is normally closed off from a hot zone 5. Following the hot zone 5 is a cold zone 6 which in turn is followed by the exit zone 7.

A reducing gas such as dissociated ammonia or hydrogen is introduced into the furnace 1 through a gas inlet 8 and the gaseous reaction products are removed from the furnace through the outlet 9 in the hot zone 5.

The moisture content or dew point of the furnace atmosphere is measured by a pair of humidistats 10, one of which is located at the beginning of the hot zone 5 while the other is located at the end of the hot zone.

Normally, the hot zone is divided into three heating sections with each section being individually heated and controlled by conventional electrical heating equipment so that the temperature of the powder passing through the hot zone can be varied throughout the three sections. The cold zone 6 is also divided into a pair of cooling sections, a water cooled section, which immediately follows the third heating section of the hot zone, and an air cooling section which precedes the exit zone 7.

As an example of the process of the invention, atomized steel powder having the following composition in weight percent was passed through the annealing furnace at a speed of 3.16 feet per minute:

	Percent
Carbon	0.08
Manganese	0.27
Phosphorus	0.012
Sulfur	0.023
Silicon	0.028
Hydrogen loss	0.80
Iron	Balance

The temperature of the steel powder and the dew point of the furnace atmosphere throughout the length of the furnace are shown in the drawing.

Dry dissociated ammonia was supplied to the furnace and the steel powder was heated to a temperature, as shown in the drawing, of about 1740° F. in the first heating section, and this temperature was gradually reduced through the second and third heating sections to a value of about 1525° F. at the end of the hot zone 5. The dew point in the first section of the hot zone 5 was approximately 140° F. and decreased progressively to a value of about 75° F. in third section of the hot zone.

Following the annealing, the steel powder was water cooled in the first section of the cold zone 6 and the temperature was reduced to a value of about 360° F. Subsequently, the powder was air cooled in the second section of the cold zone which further reduced the temperature of the powder to about 160° F. at the exit zone 7.

Following the heat treatment, the steel powder was hammermilled to restore the as-atomized particle size of the powder.

The steel powder had an apparent density of 2.95 grams/cc., a pressed density at a compaction pressure of 30 tons/sq. in. of 6.75 grams/cc., and a green strength of 4000 p.s.i. without lubricant after pressing at 30 tons/sq. in.

By maintaining the dew point of the furnace atmosphere within precise limits during the annealing process, a complete reduction of the carbon content is provided with a minimum gas flow and the possibility of the steel particles welding or permanently bonding together during the annealing is eliminated. By eliminating welding of the particles, the as-atomized particle size of the powder can be readily restored after annealing and this results in a soft compressible powder and improved density for the resulting compacted and sintered part.

Various modes of carrying out the invention are contemplated as being within the scope of the following claims particularly pointing out and distinctly claiming the subject matter which is regarded as the invention.

I claim:

1. In a method of forming steel powder, the steps of passing atomized steel powder through a heating zone, heating the powder to a temperature in the range of 1450° F. to 2100° F. in said heating zone, contacting the powder with a substantially dry reducing gas in said heating zone, the active gaseous constituents of said reducing gas comprising at least 95% by volume of hydrogen and the remainder of said volume being substantially all nitrogen, and maintaining the actual dew point of the atmosphere in the heating zone at a value below the equilibrium dew point for the temperatures involved throughout the length of said heating zone, said heating serving to soften the powder and reduce the carbon content and provide a cake-like structure to thereafter substantially restore the as-atomized particle size of the steel powder.

2. The method of claim **1**, wherein the actual dew point is maintained at a value of about 5° F. to 100° F. below the equilibrium dew point by controlling the rate of flow of reducing gas through said heating zone.

3. The method of claim **1**, wherein the reducing gas is selected from the group consisting of hydrogen and dissociated ammonia.

4. The method of claim **1**, wherein the reducing gas is passed through the heating zone in a counter-current direction to the travel of said powder through the heating zone.

5. The method of claim **1**, wherein the temperature of the powder is progressively reduced within said range through the length of the heating zone.

6. In a method of forming steel powder, the steps of passing atomized steel powder having a carbon content less than 0.20% by weight through a heating zone, heating the powder to a temperature in the range of 1450° F. to 2100° F. in said heating zone, contacting the powder with a dry reducing gas in said heating zone, said reducing gas being substantially completely dry and having a hydrogen content of at least 95% by volume of the active gaseous constituents of said reducing gas and the remainder of said volume being substantially all nitrogen, maintaining the dew point of the atmosphere at the beginning of said heating zone at a first value in the range of 100° F. to 140° F., maintaining the dew point at the end of the heating zone at a second value below 80° F., said heating serving to soften the powder and reduce the carbon content and provide a cake-like structure, cooling the powder, and thereafter breaking up the cake-like structure to substantially restore the as-atomized particle size of the steel powder.

7. The method of claim **6**, and including the step of progressively reducing the dew point from the first value to the second value throughout the length of said heating zone.

8. The method of claim **6**, wherein said second value is in the range of 60° F. to 80° F.

9. The method of claim **6**, wherein said powder is heated to a temperature in the range of 1600° F. to 1800° F. at the beginning of the heating zone and the temperature of the powder is reduced to a value in the range of 1400° F. to 1600 F. at the end of said heating zone.

10. The method of claim **1**, wherein the actual dew point is maintained at a value of about 5° F. to 40° F. below the equilibrium dew point at the beginning portion of said heating zone and said actual dew point is maintained at a value of about 60° F. to 100° F. below the equilibrium dew point at the end portion of said heating zone.

11. In a method of forming steel powder, the steps of passing steel powder through a heating zone, heating the powder to a temperature in the range of 1450° F. to 2100° F. in said heating zone, contacting the powder with a reducing gas in said heating zone, and continuously maintaining the concentration of H_2 of the atmosphere in the heating zone at a value above the $H_2O:H_2$ equilibrium value for the temperatures existing throughout the length of said heating zone, to thereby soften the particles and prevent welding of the particles together.

12. The method of claim **1** or **6**, wherein the dry reducing gas has a dew point below −40° F.

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