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FERROUS POWDER METALLURGY

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This invention relates to ferrous powder metallurgy, and particularly contemplates improvements in the manufacture of steel objects by processes which involve the usual steps incident to powder metallurgy and certain other additional steps. The invention particularly has for its object the production of a powder which is particularly suited to the manufacture of steel objects by powder metallurgy.

That the production of steel objects by powder metallurgical processes is attractive scarcely needs to be suggested; but if such suggestion were necessary, it can be readily found in the multitude of patents which have already been taken out in this field. That such processes have not been practicable is equally demonstrated by the relatively low quality of steels heretofore produced by such processes and by the limited use which is made of processes which have heretofore been proposed.

The most obvious solution to the problem of producing steel objects by powder metallurgy would appear to be the direct method of obtaining a steel powder, pressing it to the desired shape, and sintering the same. This method is not practical, however, because steel particles do not readily stick together, even with unusually high pressures employed in pressing. Part of this may be due to the fact that the surface of the particles tends to become work-hardened in the process of their obtention. It is well known that work-hardened surfaces do not readily adhere in the pressing operations.

Another method which has been employed without considerable success involves the admixture of carbon particles with iron, pressing and sintering for a prolonged period of time in order to effect even distribution of the carbon. It has been found that this method does not accomplish even distribution of the carbon, unless foreign substances such as manganese, sulfur or phosphorus are also added along with the carbon. This expedient vitiates the largest single advantage of the production of steel objects by powder metallurgy, namely, control of the quality of the finished product and exclusion of the impurities that are ordinarily encountered in the conventional steels.

A modification of this procedure employs iron powder produced from iron carbonyl. While iron carbonyl powders are rarely, if ever, completely free from either carbon or oxygen, in practice it has been attempted to overcome this difficulty by the admixture of two powders, one rich in carbon, the other in oxygen, in an attempt to

eliminate the excess of one or the other in the finished product by the chemical reaction of the oxygen and carbon at the high temperatures involved in the sintering operation. Altho large ingots weighing several tons have been produced by this method, it has been found that carbon tends to segregate at the core of the body produced even though sintering has been carried out at prolonged times and at elevated temperatures, as for example, 1200° C. and six hours.

Still another method which has been proposed suggests the decarburization of steel particles containing somewhat more carbon than the steel which it is desired to produce. This method has produced reasonably satisfactory steel bodies, but has been found difficult to control. Extremely careful control of the decarburizing atmosphere must be maintained at all times because of the inherent impossibility of maintaining all of the steel particles of exactly the same dimensions, and since the decarburization tends to produce a definite decarburization from the outside of the particle in, the result is that the smaller particles are wholly decarburized while the larger particles are only partially decarburized, and when sintering takes place segregation results when this method is practiced, just as is the case when pure carbon is mixed with pure iron.

It has also been proposed to incorporate carbon in an iron alloy produced by powder metallurgy by the employment of a brittle carbon-containing material, usually one of the ferro-alloys. This expedient has the primary disadvantage of requiring the employment of another alloying material whether desirable or not, and in addition, like some of the previously mentioned expedients, it does not produce a homogeneous body even upon prolonged soaking at elevated temperatures. These disadvantages are substantially all eliminated by the proposed new method by the application of the different expedient.

The present invention involves the production of a steel powder by any desired method, several of which are illustrated below, which powder contains a slightly greater amount of carbon than it is desired to have appear in the finished product. The steel powder is then plated with a relatively thin film of iron, or if it is desirable to incorporate other metals in the finished product, certain of these may also be incorporated in the body by plating the steel powder, as, for example, the powder may be simultaneously plated with both iron and nickel. The plated powder is washed to remove plating salts, pressed

into the form of a coherent body, preferably to the same shape as the desired material, and sintered in a neutral atmosphere to provide a dense homogeneous steel body of uniform composition and high strength. The steel powder which is plated may be obtained in several suitable manners. For example, a massive steel body may be comminuted to the desired powder form. Suitable powder may be obtained by the use of the metal spray gun, so controlling the gun and its operation that the steel, instead of being deposited in a solid mass upon some base material, is caused to be discharged into an unrestricted space where the steel particles are allowed to cool and solidify before settling. Numerous other methods for producing powder from massive bodies are also well known, but since they form no part of the present invention they will not be described here.

Again, the steel particles may be produced by suitably carburizing iron powder, which again may be obtained either electrolytically or by the reduction of an oxide of iron. If the method of reducing iron oxide is employed, it has been found that the reduction of the oxide and the carburization of the reduced iron can be carried out together by passing a carburizing of reducing gas over a heated mass of oxide particles. Suitable gases are preferably mixtures of hydrocarbons, hydrogen and carbon-monoxide, such as may be readily encountered in domestic city gas supplies.

Of the two specified sources of steel supply, the comminuted metal is somewhat to be preferred because of the solidity of the particles and the ease of handling. However, it has been found that the reduction method is measurably cheaper, and with careful control entirely satisfactory results have been obtained. When the steel powder has been prepared, it is next plated, and for this operation a barrel type plating device is much to be preferred. This device consists of an open rotating tumbling container in which the container forms the cathode, while the anode consists of a body suspended in the liquid within the container. As the container rotates, the powder particles beneath the surface of the plating liquid are quite uniformly coated with the plating metal. Satisfactory results have also been obtained by mechanically plating steel particles, as by tumbling them in a rotating drum containing either iron dust or iron balls. The soft iron tends to coat the steel particles, or in the case of the use of iron balls there is a tendency for the steel particles to wear the balls. If a proper balance is maintained between the size of the balls and the size of the steel particles, the relative hardness of the steel and iron and the speed of rotation of the mill, comparatively little comminution of the steel particles takes place, although of course if it is desired to reduce the size of the steel particles, this also can be accomplished in this operation. If the plating has been carried on by the electrolytic method, the next step is to wash the powder thoroughly in order to remove all traces of plating salts. It is desirable to press the powder while traces of moisture still remain, although this is not at all necessary for satisfactory results.

Extensive pressure is employed, to five or even twenty-five or more tons per square inch. A part of the pressure may be reduced if mechanical working of the pressed body is contemplated after sintering takes place. The pressed bodies are then baked, if at all moist, in order to avoid

puffing, and then transferred to the sintering furnace. Sintering is carried out in a neutral atmosphere at from 900 to 1250° C. If a lower temperature is employed it is apparent that a longer sintering time must be employed. Great care must be exercised in choosing the atmosphere in which the sintering is carried out, since certain gases which may normally be considered as inert exercise a very significant effect upon the pressed bodies. This is especially true during the initial stages of the sintering when the relatively infinite surface of the minute particles is exposed to the gas. The surface exposed in a pressed body to the circumambient gas may well amount to several thousand times that of the finished body. Nitrogen, for example, under conditions favorable for such reaction, tends to form nitrides in the steel, and while the nitrified surface on the exterior of the finished body may be desirable, it is certainly not desirable to have the nitrogen diffused all through the body, as might well be the case if the initial heating conditions are favorable for the taking up of nitrogen on the surface of the steel particles within the body. Similarly, hydrogen may have a slight decarburizing and embrittling action—this latter especially if any of the hydrogen is retained within the pores of the body. Again, carbon monoxide may have a carburizing effect which would not necessarily be desirable if sufficient provision had already been made in the pressed body for the desired carbon content. The ideal atmosphere is, of course, a relatively perfect vacuum, and such atmosphere is advocated if it does not unduly add to the cost of the product. Even then it is desirable to carry out a preliminary heating in vacuum in order to remove the last traces of absorbed gases from the body.

Subsequently pressed bodies may be transferred to another controlled atmosphere for completion of the sintering operation. In general it may be said that any furnace suitable for carrying out bright annealing of ordinary steel is satisfactory. As a final criterion of the suitability of such a furnace, it is best to compare the results obtained on identical pressed bodies with the furnace which it is desired to use and a vacuum furnace. If there is no appreciable improvement in the finished product obtained from the vacuum furnace, it may be said that the controlled atmosphere furnace can be depended upon to give satisfactory results.

When the sintering has largely effected suitable shrinkage in the pressed body, then it may be removed and forged at approximately forging temperatures for the particular steel in hand, in order to effect complete removal of the pores. If, however, the finished shaped body is one which does not lend itself suitably to such mechanical working, then the lack of forging can be overcome by a more prolonged sintering at elevated temperatures. In any event, it is advisable after the forging has been completed to subject the body to a reheating in order to remove forging strains.

Further treatment of the finished body depends entirely upon the character of the steel, since by the completion of the sintering and forging operations a steel body is produced which is entirely comparable to a steel forging obtained in the normal way.

As an example of the invention set forth above, steel dust obtained from steel grit used in grit-blasting is segregated from the steel grit itself and purified by washing with a strong alkali, since

in most cases a certain amount of silica contaminates this source of steel powder. The resultant powder is sieved and that which does not pass through 100-mesh sieve is rejected. The powder is then transferred to a barrel plater, and plating is carried out until approximately 7 per cent increase in weight has been obtained. After several washings in distilled water the powder is pressed at 27½ tons per square inch into bars approximately 1" square and 8" long. The bars are then dried in a flowing stream of hydrogen at gradually increasing temperatures up to about 250° C. The resultant pressed bar is then transferred to a vacuum furnace and heated by resistance to a bright yellow heat for about ten minutes. When the bar has cooled down, it is removed from the furnace and reheated in a flowing stream of hydrogen to forging heat and lightly forged to compact the same. The finished bar is wholly susceptible to tempering and an analysis shows that there is no significant variation in carbon content throughout the bar.

According to another example, pickling liquor is carefully neutralized to precipitate iron oxide and the resultant oxide washed to remove soluble impurities. The resultant oxide is dried and reduced at around 900° C. in a dried atmosphere composed of two parts cracked ammonia gas and one part domestic gas obtained by by-product coke-oven operation. This operation was carried on for approximately five hours, when samples of the resultant iron powder showed an average carbon content of .75. The powder was then transferred to a barrel plater and plated with a mixture of iron and nickel until approximately 20 per cent increase in weight has been determined. The proportion of iron and nickel plated were so controlled that about four parts of iron and one part of nickel were deposited.

The remaining operations were carried out in the same manner as in the previous example, except that after the ten-minute high temperature sintering, the sintered bars were permitted to soak for two hours in an atmosphere of dry cracked ammonia gas at about 1000° C. in order to effect more complete fusion since the amount of plated metal was relatively greater. The resulting bars showed an average composition of approximately .6 carbon and 4 per cent nickel. The strength was entirely satisfactory and the ductility somewhat greater than a similar steel produced in the conventional manner.

What we claim as new and desire to protect is set forth in the following claims:

1. Process for making metal powder suitable for conversion by powder metallurgical methods into steel bodies, which comprises providing steel

powder having a slightly greater carbon content than desired in the finished steel body, and plating onto the said powder a layer of iron in sufficient amounts to bring the average carbon content of the composite body down to the desired figure.

2. Process for making metal powder suitable for conversion by powder metallurgical methods into steel bodies, which comprises providing an oxide of iron, treating said oxide at elevated temperatures with a carboniferous reducing gas to deoxidize the same, and to leave a residual carbon content slightly greater than that desired in the finished steel body, plating ferrous metal onto the carburized, reduced powder, to reduce the average carbon content of the powder to the desired figure, and washing the powder to remove undesired soluble substances.

3. Process for the metallurgy of iron to form steel bodies which comprises passing a carbonaceous reducing gas over iron oxide to reduce the oxide and leave a slightly larger amount of carbon in the metal powder than is desired in the finished steel product, depositing by electrolysis on the powder, iron and other metals, to bring the average analysis of the powder to the average analysis desired in the finished sintered body, removing any soluble matter from the plated powder, pressing the same to the desired shape, and sintering the shaped body in a neutral atmosphere.

4. A shaped metal body consisting of a compacted mass of carboniferous iron particles, each of which carries an electrolytically deposited surface of iron and other elements commonly found in steel.

5. In the art of ferrous powder metallurgy, the improvement which comprises coating steel particles with a non-carbonaceous ductile constituent of steel, pressing the particles to a desired shape, sintering the shape in a neutral atmosphere suitable for bright annealing steel, hot-working the sintered shape and removing the hot-working strains by a further heat-treatment.

6. In the art of ferrous powder metallurgy, the steps which comprise purifying carboniferous ferrous particles containing undesired impurities, to remove such impurities, depositing on their surface a non-carboniferous surface of ductile constituents of steel to produce powder of desired final analysis, pressing the powder to desired final shape, and maintaining the pressed bodies at an elevated temperature for a length of time sufficient to produce in the bodies the desired dense metallurgical structure.

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