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METAL POWDER

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4 Claims. (Cl. 75—28)

This invention relates to the production of steel powder, more particularly to an improved metal powder, produced from cheap source materials and available for powder metallurgy and other purposes. This application is a division of my prior application, Serial No. 413,367, filed October 2, 1941.

In the production of stainless steel, and particularly in the alloy steel casting industry, there is a considerable amount of borings, turnings and grinding residues available which contain not an inconsiderable percentage of stainless steel. These residues are produced, for the most part, by grinding stainless steel or other high alloy castings with an abrasive wheel. The product or grinding residues produced is in quite finely divided form, but due to its contamination with the abrasive and bonding material of the wheel, it has heretofore attained no commercial market.

In the field of power metallurgy, stainless steel powder in various meshes has a considerable potential use. It has been found, as is more particularly explained in my copending application, Serial No. 413,367, that this powder may be compacted under suitable pressures to produce a self-sustaining compact which, with the proper technique, may be sintered to produce an ultimate article of very satisfactory physical characteristics.

As explained in that prior application, the stainless steel powder entering these compacts may economically be produced from stainless steel scrap by suitable heat treatment followed by intergranular corrosion, ball milling or other disintegration treatment and classification.

It has now been found that a particularly useful and economical source material for stainless steel powder or other high alloy iron powders are the swing grinding residues produced by the final surface of alloy casting and similar alloy steel objects.

In the past this raw material, due to the relatively high percentage of contaminated material, has had no commercial outlet. It has been found that this material may be treated by simple methods to produce a stainless steel or other alloy powder in desirably finely divided form and free from associated contaminants. The product thus produced is particularly useful in certain fields, such as in the spraying of metal powder by the Schoop gun, in which very fine material of the order of -325 mesh is preferred.

The raw material which is subjected to treatment according to the present invention consists

of grindings, borings, turnings, which contain the usual contaminants. This material contains quite a large percentage of such products as silica, silicon carbide, organics (from the bonding agent of the grinding wheels), and the like. In most circumstances this material also contains quite a high percentage of ordinary iron, and sometimes brass.

In carrying out the invention this type of raw material is first heat treated in a special thermal range and under conditions which conduce to the precipitation of carbides in the grain boundaries and which coincidentally eliminates certain of the contaminants. In this step of the process the raw material preferably is heat treated in a rotary kiln furnace between 500° and 900° C. for a period varying from ½ to 24 hours. Preferably this heating is done cyclically, that is to say, the temperature is cyclically elevated and lowered within the carbide precipitation range to accelerate carbide precipitation. Due to this heat treatment carbides are precipitated in the grain boundaries and slip planes; coincidentally, a considerable amount of contaminants such as sulfur and the like are burnt off.

The product produced by the heat treatment is then quenched in a suitable contact corroding medium such as in a spent pickling liquor, from steel pickling, to which liquor has been added of the order of 5% of sodium fluoride and sometimes up to 5% sulphuric acid. In thus quenching the hot material a considerable amount of the adherent abrasive is detached from the metal particles by exfoliation. Due to the contact with the pickling liquor additional amounts of the abrasives are detached by reason of the undercutting effect of the pickle. Since the heat treated material is quenched in the pickle liquor the temperature of the latter is quickly elevated and the pickle is extremely active.

After the quenched product has been retained in the pickle liquor for a predetermined period of time, usually from about 30 minutes or more, the liquor is separated from the solid residue. This may be done in any suitable continuous filtering apparatus or by decanting off the supernatant pickle liquor.

The residue from the pickling stage may be washed to remove adherent pickle and is then screened. It has been found that by a simple screening method a very large proportion of the oxides and abrasives present in the original residue can be removed. Specifically, it has been found that most of these oxides and abrasives

are separated from the metallic particles by screening through approximately a 100 or 200 mesh screen and discarding the -100 or -200 mesh material. It has been found, similarly, that much of the low iron alloy content in the original residue will be removed in this screening operation, due to the fact that the low alloy iron is corroded to fines during the pickling, whereas the substantially corrosive-resistant stainless steel particles are unattached.

The coarse material produced from the above screening operation comprises essentially stainless steel or other high alloy iron powder and is then disintegrated. This may effectively be done by crushing the material in a suitable type of hammer mill and preferably in a micro-pulverizer. This type of machine, as is known, can be set for a more or less definite degree of disintegration, i. e., for -100, -200, and -300 mesh material. The micropulverizer is peculiarly effective for this operation, for the material can be disintegrated at the rate of about 25 to 400 pounds per hour. In this type of apparatus, as those skilled in the art recognize, a series of stellite hammers move through a small distance, of the order of $\frac{1}{2}$ of an inch, at extremely high speed. It will be understood that in this disintegrating operation the material to be treated may be fed to the pulverizer either wet or dry or in admixture with any suitable lubricant. During this disintegrating operation, due to the cold work applied to the metal, the alloys treated (18-8; 18-8-Mo.; 18-8-Ti.; etc.) are rendered magnetic.

The crushed material produced in the pulverizer step may then be treated in a suitable corrodent, so as to selectively attack the grain boundaries (high in carbides) and for removing imbedded abrasive scale, low alloy iron and the like. The corrodent for this step of the process may comprise a suitable aqueous solution of acid ferric sulphate and sodium fluoride. The period of retention in the corroding solution will vary depending upon the particular product treated, the temperature of the corroding solution, and the like.

The crushed and corroded material is now removed from the corrosion vat and is washed to free it of adherent corrodent. The product is then concentrated, preferably on a Welfley table, to separate the metal particles from the non-metallic particles, i. e., abrasive material, oxides, carbides and the very fine unalloyed iron. These latter products are discarded in the tailings and the concentrate is recovered, which contains of the order of 90 to 98% of stainless steel powder.

If desired, the tailings, middlings and concentrate produced in the above concentration step may be further concentrated either by flotation, electrostatic or magnetic separation.

The final concentrate, which comprises essentially an 18-18 steel powder, is then contacted or washed with a hot nitric acid solution between about 15% and 20% concentration. This treatment serves a number of useful functions, namely, to brighten or shine such powder. The material thus produced, as noted previously, is particularly useful for spraying in metal guns of the type mentioned because of its desirably fine mesh, and for such purpose the product can be used directly.

The product may also be used for powder metallurgy purposes. However, for this use it is necessary to subject the material to an additional treatment, preferably to a dry hydrogen anneal

in a temperature range between about 900° C. and 1200° C.

As will be appreciated by those skilled in the art, the heretofore waste material, namely, the grinding residues, may be processed to produce valuable alloy steel powder by modifications of the above method. Useful discrete steel powder may be produced from the described source material by first crushing the raw material in a micro-pulverizer or similar machine, to such a degree that it passes through a 200 mesh screen. Since these metal particles are already quite severely work hardened (due to the grinding and boring operations which produce them) they are quite brittle and are readily disintegrated. During this grinding operation the contaminants in the raw material, i. e., grinding oil, sulphur, rubber, Bakelite and the like, serve beneficially in the role of lubricants. During this grinding operation the attached or adhered abrasive particles are largely removed from the mesh by impact.

The ground product described above is then fed to a screen which classifies the material into two fractions, one of a screen analysis between 200 and -325 mesh and the other a -325 mesh. These two fractions are then each passed through a magnetic separator, or if desired each fraction may be treated by gravity separation methods such as flotation, Welfley table separation, and the like.

The magnetic concentrate, or the concentrate produced by table or flotation, is treated with a nitric acid solution of suitable concentration. This treatment serves to degrease the product by undercutting and to passivate the stainless steel. If the product is to be used for a remelting addition agent, in lieu of passivation, it may be treated with a typical pickling solution which serves to dissolve the sulphur and levigate the extreme fines of abrasives, oxides, carbides, Bakelite, and the like. This scum may then be removed from the top of the pickle bath.

The material thus produced is washed and dried and may be briquetted in any suitable manner, so as to be employed in a high alloy steel melt.

If the product is to be used as a pigment, for example in paints, it is preferably ball milled either in a wet ball milling operation in contact with a liquid medium such as relatively low boiling petroleum oil fractions of the type of Varsol or it may be dry milled and polished with stearic acid.

It is to be observed that if the product from the magnetic separator or other concentrator is too high in iron the nitric acid treatment or the pickling is continued so as to dissolve ordinary iron and steel.

If the material produced is to be used for spraying, it may be mixed with a flux or with a lower melting such for example as copper. Excellent results have been attained by mixing an 18-8 powder of the type described above with from about $\frac{1}{2}$ to 2% or more of copper powder. In these circumstances it is found that the sprayed metal coating quickly builds up on the sprayed base and is characterized by an extremely high adhesion and tenacity. Another particularly beneficial addition agent for this purpose is the eutectic powder described in my copending application, Serial No. 414,037, filed October 12, 1941. A particularly effective method of homogeneously incorporating the flux or the low melting powder in the stainless steel is to run the

mixture through the micro-pulverizer. Another method is to separately grind the flux or low melting point metal powder so that its particle size will be equal to and preferably less than that of the stainless steel powder, and then mixing the two. For 18-8 spraying powders, in addition to copper and the iron eutectic powder mentioned the low melting calcium-manganese-silicon alloys, silver-copper-phosphorus eutectic alloy, ferro-manganese and the like, may be utilized. Any metallic product compatible with stainless steel powder and which serves to accelerate or facilitate the bonding of the sprayed powder to the base material may be used.

After spraying the improved powder on the base material, for example, on a low carbon steel base, the sprayed coating may be wire brushed to secure a desired matte finish, or the material can be abraded flat and then brushed, and if desired finished by polishing. Similarly, the sprayed material may be electrolytically polished.

In some circumstances, where an extremely tenacious bond between the sprayed surface and the base plate is desired, it is advantageous to anneal the article under bright annealing conditions.

Spraying powder of the type above described is useful in a wide variety of fields, as, for example, in the application of corrosion-resistant, relatively fine-sectioned surfaces to more corrodable base metal, and to the building up of steel surfaces, by the spraying technique, to a desired specification. In the event that highly wear-resistant coatings are desired, the applied sprayed coating may be suitably treated, as by flame hardening, or high frequency hardening, to improve the wearing qualities of the applied coat.

I claim:

1. A method of producing stainless steel powder from contaminated residues containing stainless steel which comprises crushing the material in a suitable mill to produce a powder material of approximately -200 mesh and to work harden the metal content; screening the dis-

integrated product to produce a fraction of between +200 and +325 mesh and a fraction of -325 mesh; subjecting the fractions to magnetic separation to recover a metallic magnetic concentrate and subjecting the concentrate to the action of a solution which passivates the stainless steel.

2. A method of producing stainless steel powder from contaminated residues containing stainless steel which comprises crushing the material in a hammer mill to disintegrate and work harden the metal, screening the crushed material to produce a fraction between about 200 and 325 and a fraction of -325 mesh, subjecting the fractions to magnetic separation to recover a metallic concentrate and subjecting the concentrate to the action of a nitric acid solution.

3. A method of producing remelting material from contaminated residues which contains stainless steel which comprises crushing the material in a micro-pulverizer to produce a powdered product of approximately -200 mesh and to work harden the metal content, screening the disintegrated product to produce a fraction of between 200 and 325 mesh, subjecting the fractions to magnetic separation to recover a metallic concentrate and subjecting the concentrate to a pickling solution which degreases and passivates the steel particles and briquetting the cleaned steel.

4. A method of producing stainless steel powder from contaminated residues which comprises crushing the material in a micro-pulverizer to produce a powder material of approximately -200 mesh, screening the crushed product to produce a fraction of between 200 and 325 mesh and a second fraction of -325 mesh, subjecting the fractions separately to magnetic separation to recover metallic concentrates, subjecting the concentrates to the action of a nitric acid solution to clean and passivate the steel and for a period sufficiently long to dissolve contained iron, washing the cleaned stainless steel and then annealing the cleaned steel in dry hydrogen.

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