TREATMENT OF METAL POWDER

Filed Dec. 2, 1959 2 Sheets-Sheet 1

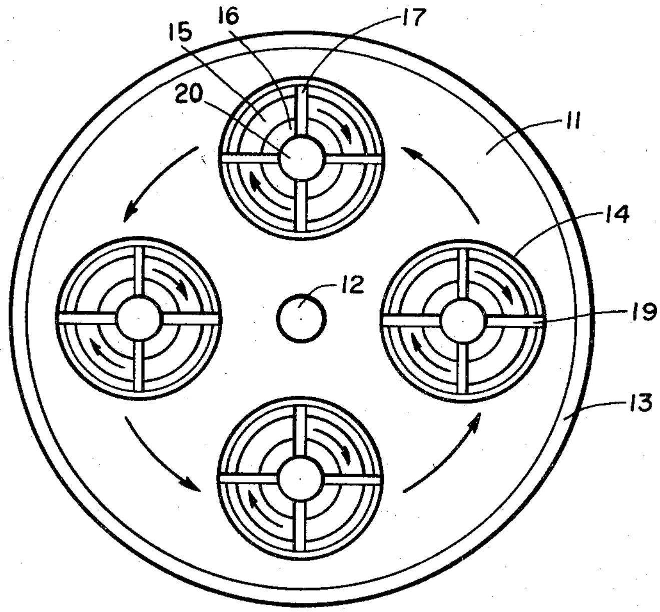

FIG. 1

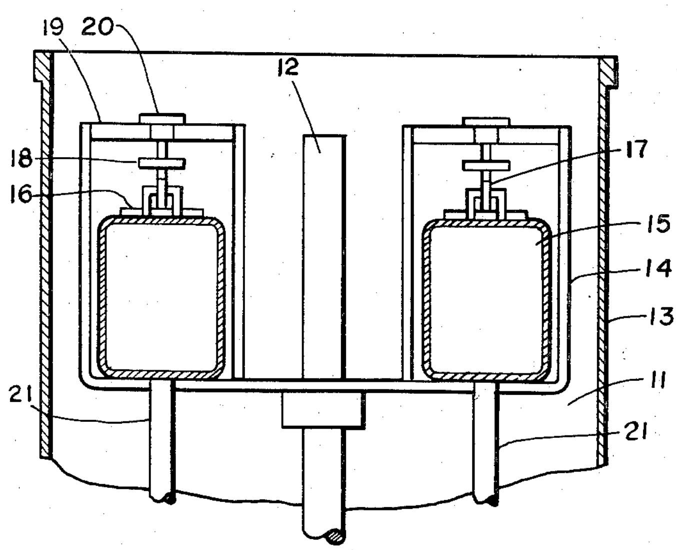

FIG. 2

DEWITT H. WEST
DAVID M. LLEWELYN
INVENTOR.

BY [signature]

ATTORNEY

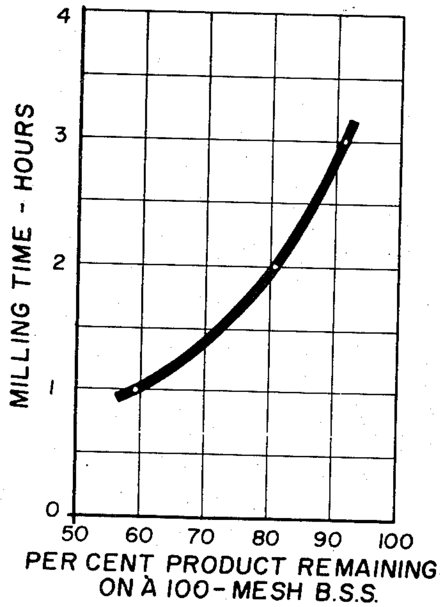

FIG. 3

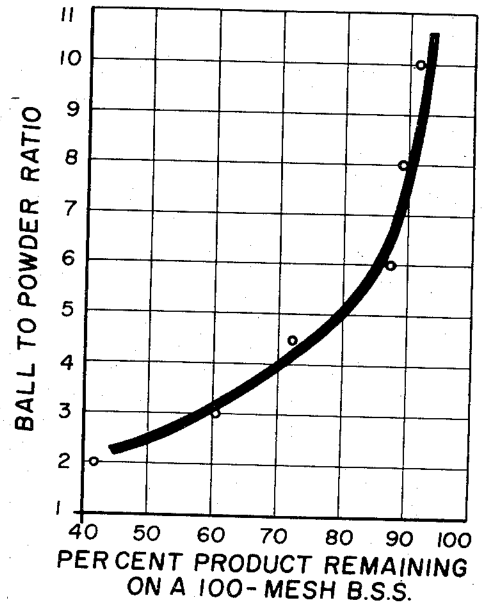

FIG. 4

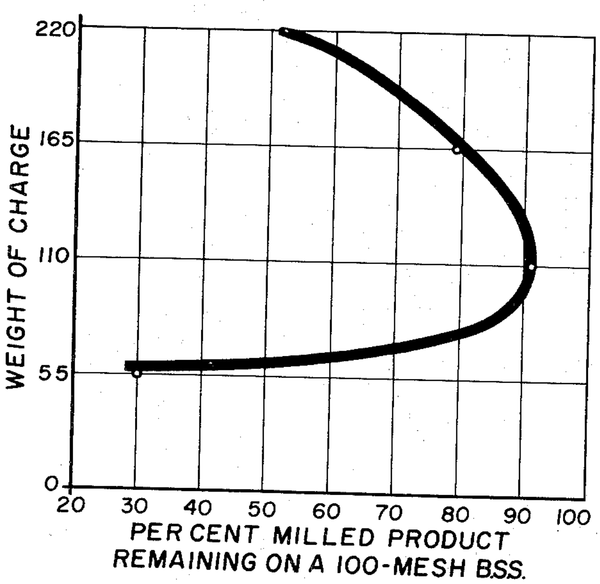

FIG. 5

DEWITT H. WEST
DAVID M. LLEWELYN
INVENTOR.

BY Q. Q. Deller

ATTORNEY

2,995,780
Patented Aug. 15, 1961

2,995,780

TREATMENT OF METAL POWDER

De Witt H. West, Port Eynon, and David M. Llewelyn, Cwmdwr, Clydach, England, assignors to The International Nickel Company, Inc., New York, N.Y., a corporation of Delaware

Filed Dec. 2, 1959, Ser. No. 856,754
Claims priority, application Great Britain Dec. 9, 1958
2 Claims. (Cl. 18—48)

The present invention relates to the mechanical treatment to agglomerate soft metal powders of very fine particle size and, more particularly, to a method for milling fine metal powder such as nickel powder so that the particles thereof will coalesce and grow in size.

It is well known that metal powders of very fine particle size are extremely difficult to handle, use, and/or package. This is particularly true whenever metal powder is employed as an addition in an alloying process.

It is also well known that the flowability of metal powders is an important factor wherever large-scale powder metallurgical manufacturing processes depend on rapid filling of the molds, as, for example, in the manufacture of porous bearings or machine parts. Since feeding is ordinarily accomplished by volumetric measure, rapid and uniform filling with the elimination of pockets in the die cavity or in the feeding device is of great importance. Thus, if the metal powder has superior flow characteristics, it is suitable for high-speed compacting equipment. Conversely, if the powder has poor flow characteristics, an uneconomical feeding results. One of the methods used in the art to abate the poor flowability of powders is to add equipment such as vibrators to the feed mechanism. However, this entails additional expense and increases the cost of the article formed from the powders.

Although many attempts were made to overcome the foregoing difficulties and other disadvantages, none, as far as we are aware, was entirely successful when carried into practice commercially on an industrial scale.

It has now been discovered that powders of fine particle size coalesce and agglomerate when subjected to mechanical stresses under controlled conditions, and that the flowability of such treated powders is greatly enhanced.

It is an object of the present invention to provide an inexpensive, readily available method for increasing the particle size of fine powders and their flowability by the application of external mechanical forces.

Another object of the invention is to provide a process for coalescing carbonyl metal powders by external mechanical stresses without the use of binding and/or agglomerating agents to produce discrete, granular particles of larger size.

The invention also contemplates providing a powder of controlled minimum particle size.

Still another object is to provide a granular powder having superior flowability properties.

Other objects and advantages will become apparent from the following descriptoin taken in conjunction with the accompanying drawings in which:

FIGURE 1 is a plan view of a high-speed ball mill;

FIGURE 2 is a front view of only 2 containers of the ball mill of FIGURE 1;

FIGURE 3 is a graphical representation showing the effect of milling time upon particle size;

FIGURE 4 depicts a graph of ball size plotted against particle size; and

FIGURE 5 illustrates the effect of the total weight of the charge on the particle size of the product.

Broadly stated, this invention contemplates and relates to the treatment of relatively soft metal powder exemplified by nickel powder produced by the decomposition of nickel carbonyl and commonly known as carbonyl nickel powder, carbonyl iron powder of low carbon content, copper powder, and aluminum powder. The powders to which the invention relates are those of very fine particle size, i.e., less than about 8 microns. According to this invention, relatively soft powder is subjected to sufficient translational and rotational forces while being simultaneously peripherally restrained to cause movement of the powders in a direction toward the peripheral restraint. That is, the powders are charged into a confining means or other receptacle containing grinding balls, and said charge, comprising balls and powder, is subjected to a first acceleration of at least about 1 gravity induced by rotation of the confining means about its axis while substantially simultaneously subjecting the charge to a second acceleration. This induced second acceleration is at least 2 times greater than the acceleration induced by rotation of the mill about its own axis and the second acceleration, which is advantageously about 2 to about 12 times greater than the first induced acceleration, is applied in a direction that is substantially perpendicular to the axis of the confining means. Ball milling is well known as a method of reducing the particle size of the milled product. If a soft metal powder is milled under high-energy, high-speed conditions, as shown hereinbefore, such that the powders are subjected to the combination of sufficient translational and rotational forces, the result of the milling according to the invention is most surprisingly exactly the opposite, namely, that the particle size grows. In consequence thereof, coarse powder, i.e., over 500 microns, can be produced from finer powder, i.e., powder having a particle size of from about 2 microns to about 8 microns. In addition, the coarse particles formed thereby have substantially improved flowability and a greater apparent density.

FIGURES 1 and 2 depict a suitable high-speed ball mill **11** that is provided with a main central drive shaft **12** and an outer casing **13**. A cage **14** adapted to carry a plurality of cylindrical containers or pots **15** in spaced relationship is suitably mounted to drive shaft **12**. Each pot is provided with a lid **16** which is secured to the pot by securing arm **17** and screw cap **18**. In order to hold the pots in place, an arm **19** is attached to screw cap **18** by fastening nut **20**. The pots have vertical drive shafts **21** which along with main central drive shaft **12**, are connected to a geared mechanism (not shown) that is designed to set up a planetary motion as shown in FIGURE 1. For example, while drive shaft **12** is turning the cage **14** and pots **15** counter-clockwise, shafts **21** are spinning the pots clockwise or directly opposite to the direction of rotation of main central drive. By this assembly, the cylindrical containers or pots **15** are moved as a whole through a circular path around a vertical axis (main drive shaft **12**) at high speed while they are simultaneously rotated about their own axis (shafts **21**) in the opposite direction, also at high speed. This type of movement causes a turbulent agitation of the balls and soft metal powder which results in multiple contact of the balls with the powder and the transmission of high energy by means of translational and rotational forces to the powder. As a consequence of this action, the particles charged into the mill actually grow in size and yet remain substantially discrete. In other words, the whole mass of the feed powder does not coalesce as a unit. The particles so formed are substantially granular and have a high particle density approaching that of the metal concerned. Furthermore, these agglomerated powders have good flowability as defined by the angle of repose.

In the same connection, we also find that powders of different average particle size can be made by varying the milling conditions. Thus, particle size of the feed, the weight of the charge including the balls and powder, the ball-to-powder ratio, the composition of the balls, the residence time of the charge in the mill, the speed of rotation of the mill, the energy input to the mill, alone or in any combination, with materially affect the particle size of the end product.

Thus, the particle size of the powder used affects the final size after milling, the coarser the initial powder, the greater being the average particle size of the product. Oftentimes, the particle size of the feed will be dictated by the kind of powder used. For example, carbonyl nickel power may average from 2 microns to 8 microns according to the conditions of decomposition.

In carrying the invention into practice, it is advantageous to control the variable factors of the process operations in order to obtain the desired particle size of the end product. To illustrate the effect of these variables, a series of experiments was conducted in a ball mill. Such a mill was used in the examples given as is described in FIG. 1. The ball mill used in all these tests comprised a mill container of 7 inches diameter and 8 inches axial length into which is charged the feed. The starting powder used in the examples consisted of discrete spherical particles of less than about 8 microns average size. The mill was operated so that it was moving at 190 revolutions per minute through a circular path of 6 inches radius and turning about its own axis at 100 revolutions per minute. To contact the powder the mill was charged with balls, steel balls of ⅜–½ inch diameter being used. Advantageously, the milling is carried out dry, no advantage being obtained if a liquid, e.g., an alcohol, is added, and if water is added the particle size of the final product is descreased. For example, 6 pounds of fine nickel powder previously moistened with 9% water were milled with steel balls and the ball-to-powder ratio was 3 to 1. After 2 hours of milling in the mill operated under the aforementioned conditions, the average particle diameter was reduced to less than one-half its original size.

The result of varying these conditions is shown by the hereinafter listed tables. Table I shows the effect of varying the milling time when milling with a 10:1 ball-to-powder ratio and 11 pound charge (balls plus powder) as when such a mill is used for grinding the container is usually run about half full of the charge of balls and material.

Table I

Milling Time (Hours)	Percent of End Product Remaining on a 100-Mesh B.S.S.[1]
1	59.1
2	80.1
3	91.2

[1] A 100-mesh British Standard Sieve has openings of 147 microns or 0.0058 inch.

Table II shows the effect of varying the ball-to-powder ratio when the mill is operated for 3 hours with 11 pounds charge (balls plus powder).

Table II

Ball-to-powder Ratio	Percent of End Product Remaining on 100-Mesh B.S.S.
2:1	40.5
3:1	60.2
4.5:1	71.1
6:1	87.2
8:1	89.3
10:1	91.2

From these tables FIGS. 3 and 4 are respectively plotted and both curves indicate that as either the ball-to-powder ratio and/or the time increases, their slopes become asymptotic. This demonstrates that there is an optimum milling time, and if this time is exceeded the average output of larger particle size begins to decrease per unit time. Furthermore, the higher the ball-to-powder ratio, the greater is the final average particle size obtainable, but again the output of powder of this size in unit time decreases. The probable cause of this is work-hardening, which reduces the tendency of the particles to coalesce as the powder stores part of the energy expended on it after being worked. Therefore, by reducing the ball-to-powder ratio, the amount of work-hardening can also be reduced. Thus, if the aim be the maximum output of the coarsest powder per unit time, there is therefore also an optimum ball-to-powder ratio, as is shown by the curve of FIGURE 4, wherein ball-to-powder ratios are plotted against percent product remaining on a 100-mesh British standard sieve.

As was shown by the foregoing experiments, when the milling is carried on under optimum economic conditions, only a proportion of the powder produced is of the average size required. This can, of course, be separated by sieving and it is found that the remaining under-sized fraction can be caused to grow in size by being recharged. It is also discovered that the yield when recharged powder is milled can be substantially increased if the powder is annealed between successive millings. For example, nickel powder may be annealed under hydrogen for one hour at 800° C.

To further illustrate the effects of another variable factor in order to obtain the desired particle size in the end product, additional tests were run in the same mill under the same conditions as in the previous experiments holding the duration of operation of the mill constant at 3 hours and using a ball-to-powder ratio of 10:1 and varying the weight of the charge with the results as shown in Table III.

Table III

Run No.	Weight of Charge, lbs.	British Standard Sieve			
		+30	−30 +60	−60 +100	−100[1]
1	5.5	-------	8.3	21.7	70.0
2	11.0	18.9	43.7	28.6	8.8
3	16.5	16.4	39.4	24.6	20.4
4	22.0	1.8	21.6	28.3	48.4

[1] British Standard Sieve scale wherein 100-mesh is equal to 147 microns

Using the data of Table III, Figure 5 is drawng wherein the abscissa is the percentage of powder remaining on a 100-mesh British Standard Sieve and the ordinate is the weight of the charge. The curve is parabolic showing that there is an optimum weight of charge. Therefore, the best yields of coarse particles are obtained when the total charge occupies about half the container.

As a result of these tests with the aforementioned mill operating at high speed, the optimum economic conditions for particles larger than 500 microns (+30 mesh B.S.S. scale) from a starting material of less than 8.0 microns average particle size are 3 hours milling with 11 pounds charge (ball+powder) and a 3:1 ball:powder ratio using ⅜–½ inch steel balls. Furthermore it is apparent that these conditions provide sufficient energy coacting translational and rotational forces to agglomerate the particles.

The agglomerated, discrete powders of this invention exhibit improved flow characteristics, as defined by the angle of repose. For example, the powders of Run 2, Table III, were tapped through a 30-mesh screen onto the end of a vertical cylinder having an 0.7 inch diameter. The base angle of the cone formed by such tapping was measured and found to be only 29° from the horizontal. However, when carbonyl nickel powder was identically tested, it had an angle of repose of about 66° from the horizontal. Thus, the powders of this invention have much the superior flowability. Furthermore, compacts produced therefrom are characterized by having good green strength.

However, if ⅜–½ inch steatite balls are used instead of steel balls in the mill described above, the optimum conditions are different, being in fact milling for 7 hours with 6 pounds of charge and a ball-to-powder ratio of 3:1.

The powders of the present invention are particularly adapted to be employed in general use in powder metallurgy, alloying and in the manufacture of welding electrodes.

Although the present invention has been described in conjunction with preferred embodiments, it is to be understood that modifications and variations may be resorted to without departing from the spirit and scope of the invention, as those skilled in the art will readily understand. Such modifications and variations are considered to be within the purview and scope of the invention and appended claims.

We claim:

1. A process for substantially increasing the size of soft metal particles selected from the group consisting of nickel, iron, aluminum and copper powders having a

particle size in the range of about 2 to about 8 microns comprising providing a charge of grinding balls and substantially dry, soft metal powders and subjecting said substantially dry charge within a confining means to an acceleration of at least about one gravity induced by rotation of said confining means about its axis while substantially simultaneously subjecting said charge to an acceleration of at least about two times greater than said rotationally-induced acceleration, said acceleration being induced externally with respect to said confining means, and being applied in a direction substantially perpendicular to the axis of said confining means.

2. The process as claimed in claim 1 wherein the soft metal particles are substantially dry nickel powders.

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