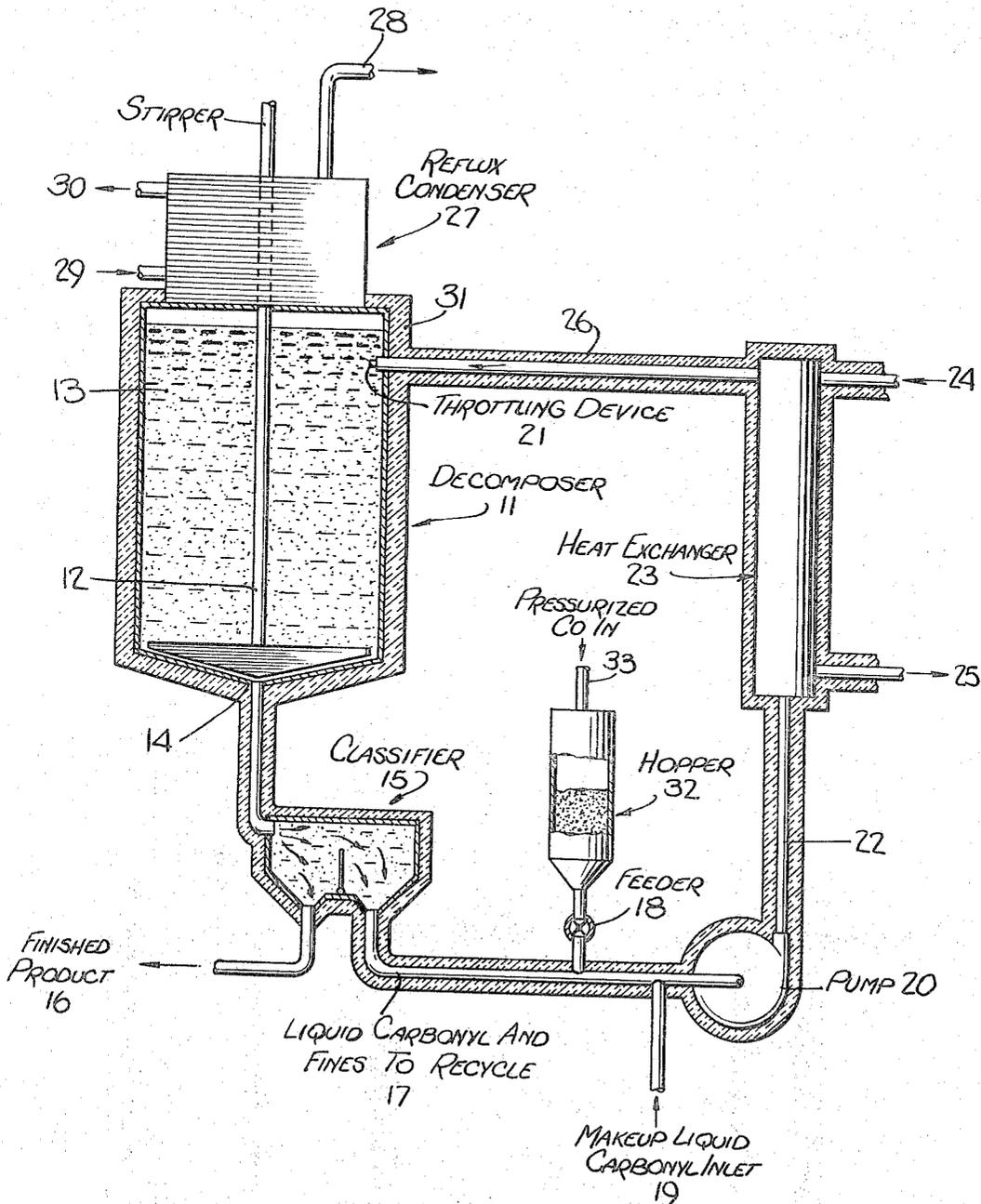


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METHOD FOR THE PRODUCTION OF METAL AND
METAL-COATED POWDERS
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METHOD FOR THE PRODUCTION OF METAL AND METAL-COATED POWDERS

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ABSTRACT OF THE DISCLOSURE

Metal carbonyls, e.g., nickel and iron carbonyls, are decomposed from the liquid state, preferably in a continuous cyclic process involving establishing a decomposing zone and a heating zone, each substantially filled with liquid metal carbonyl containing suspended particles, e.g., metal powder, pumping liquid metal carbonyl and suspended particles to the heating zone wherein heat for decomposition is supplied without boiling the liquid metal carbonyl and wherein the pressure applied to the liquid metal carbonyl is maintained higher than the pressure in the decomposing zone and injecting the heated mixture of liquid metal carbonyl and suspended particles into the decomposing zone to cause at least a local reduction in pressure whereby decomposition of the liquid metal carbonyl to metal and carbon monoxide is facilitated with plating of metal from the decomposition of liquid metal carbonyl upon the suspended particles.

The present invention is directed to a method and apparatus for producing metal powders and metal-coated powders by the decomposition of a metal carbonyl and, more particularly, is directed to a special method and apparatus for decomposing metal carbonyl at high production rates.

The decomposition of metal carbonyls from the vapor phase to produce powders of the corresponding metal and to produce metal-coated powders has long been known. Such vapor-phase carbonyl decomposition processes have been attended by production handicaps, including large and expensive equipment, the requirement for compressors and other expensive auxiliary equipment, and by a low production rate. These factors have entailed a very considerable capital expenditure for plant facilities to produce metal powder products by the carbonyl route. In conventional vapor-phase decomposition of metal carbonyls such as nickel carbonyl and iron carbonyl, the decomposition is effected in a standard one-meter diameter decomposer at essentially atmospheric pressure. Such decomposers have a relatively low production rate of about 100 pounds of metal per hour. The carbon monoxide obtained as a result of the vapor-phase metal carbonyl decomposition is recovered. In order to employ the carbon monoxide for the production of additional metal carbonyl by the carbonylation reaction with a metal such as nickel or iron, the carbon monoxide is compressed to a relatively high pressure which may be, for example, as high as 1,000 pounds per square inch gage (p.s.i.g.), since such pressures facilitate the carbonylation reaction. In order to carry out this procedure on an industrial scale, large diameter piping, large gas holding capacity, and expensive compressor equipment is required. Another disadvantage of the prior process resides in the fact that a vaporizer is required to convert the liquid carbonyl to vapor and problems are encountered in the vaporizer due to excessive plating. A further disadvantage of the vapor decomposer is that decomposition must be substantially complete, i.e., to a value not exceeding 10 grams of nickel carbonyl per cubic meter, to prevent plating and seizing

of the compressor used to compress the effluent carbon monoxide from the decomposer for reuse in further carbonylation.

Metal powders produced by the vapor-phase decomposition of metal carbonyls have many advantages, particularly from the standpoint of purity. On the other hand, for many uses in the powder metallurgy field such powders have physical disadvantages due to the particle size and shape thereof. For example, the flow properties of such powders are unsatisfactory in many instances. In addition, it is considered that in many powder metallurgy processes, composite powders comprising a metallic or nonmetallic core with a metal coating thereover would be desirable. It is difficult to produce such coated powders by conventional vapor-phase carbonyl decomposition processes since interference due to static charges and complex heat transfer problems are encountered.

In view of the foregoing factors and many others, it would be highly advantageous to provide a means for producing metal powders and metal-coated powders by the decomposition of a metal carbonyl in simpler plant and to provide metal powders and metal-coated powders having desirable physical and chemical properties, including high purity and improved flow properties at high production rates.

An improved process and apparatus have now been discovered which enable the production of metal powders and metal-coated powders by the decomposition of a liquid metal carbonyl wherein the high heat transfer rates associated with liquid materials are utilized and wherein a relatively simpler and smaller plant is employed.

It is an object of the present invention to provide a method for the production of metal powders and metal-coated powders by the decomposition of liquid metal carbonyl.

It is a further object of the invention to provide an apparatus for the production of metal powders and metal-coated powders by the decomposition of liquid metal carbonyl.

A still further object of the invention is to provide a recirculating method for the production of metal by the decomposition of liquid metal carbonyl wherein high heat transfer rates to liquid metal carbonyl are utilized.

Another object of the invention is to provide an apparatus for the decomposition of a liquid metal carbonyl on a simple and relatively small scale but, nevertheless, with a high production rate of metal.

Other objects and advantages of the invention will become apparent from the following description when taken in conjunction with the accompanying drawing which depicts an advantageous apparatus for carrying out the process contemplated in accordance with the invention.

Broadly stated, the invention comprises decomposing a metal carbonyl in a liquid environment at a temperature near but not exceeding the boiling point of the metal carbonyl at the ambient pressure under conditions such that at least a portion of the metal carbonyl being decomposed is subjected to a reduction in pressure to induce decomposition of the metal carbonyl and to provide carbon monoxide and the corresponding metal.

As is known in the art, the boiling point of liquid metal carbonyl is elevated by increasing the pressure. In the case of nickel carbonyl and iron carbonyl, which are volatile liquids at atmospheric temperature and pressure, the decomposition from a liquid environment can be carried out at atmospheric pressure and even sub-atmospheric pressure, although pressures up to about 3,000 p.s.i.g. or even about 5,000 p.s.i.g. may be employed. It will be appreciated that the temperature is varied concomitantly with the pressure to effect decomposition of the liquid carbonyl at a temperature not exceeding the boiling point. In gen-

eral, temperature conditions for carrying out the decomposition reaction can be varied over a wide range from ambient temperatures, e.g., about 70° F. or 100° F. or 150° F. up to, e.g., about 400° F. or 500° F. or 650° F. or even higher depending upon the operating pressure.

At least a portion of the liquid metal carbonyl undergoing decomposition is subjected to a decrease in pressure, which decrease in pressure may be of a small order. For example, the decrease in pressure occurring on the rearward faces of a propeller stirrer is sufficient to initiate decomposition of liquid nickel carbonyl at the boiling point thereof. The requisite decrease in pressure may be effected in other ways. For example, a portion of the liquid metal carbonyl to be decomposed may be removed from the decomposition chamber, pressurized and heated to pressure-temperature conditions above those maintained in the decomposition chamber but below the boiling point thereof, and may be injected through an opening or orifice into a body of liquid metal carbonyl at approximately the boiling point at a lower pressure. In this way, boiling of a portion of the liquid metal carbonyl undergoing decomposition is initiated and decomposition occurs rapidly under ambient conditions which are highly concentrated with respect to the metal carbonyl undergoing decomposition. Thus, in the case of nickel carbonyl, each liter contains about one pound of nickel.

It will be appreciated that decomposition of a metal carbonyl is an endothermic process and the problem to be solved in providing an effective decomposition process is that of providing a sufficient heat supply to the system wherein decomposition is being effected. This problem is effectively solved in accordance with the invention since the materials being treated are liquids having higher heat capacity per unit volume as compared to gases and having good heat transfer characteristics.

The present invention comprehends the decomposition of a metal carbonyl from the liquid state to provide the corresponding metal in particulate form and also comprehends the production of particles coated with the said corresponding metal. In the latter case, particles of material to be coated are introduced into the decomposition zone, advantageously by mixing and suspending such particles in the liquid metal carbonyl to be decomposed.

A particularly effective means for carrying out the decomposition process contemplated in accordance with the invention is depicted in the accompanying drawing which schematically presents a feed recycle or recirculating system for decomposing a liquid metal carbonyl such as nickel carbonyl on a substantially continuous basis. The apparatus essentially comprises a decomposing chamber having coupled thereto an auxiliary heat exchange means constructed such that the pressure within the heat exchanging circuit can be maintained at a higher level than the pressure in the decomposer chamber itself. In the drawing, reference character 11 indicates a decomposer chamber which advantageously is provided with stirring means 12. The chamber is adapted to be filled substantially with a liquid metal carbonyl indicated at 13. Liquid metal carbonyl bearing particulate products, including metal-coated products, resulting from the decomposition reaction are withdrawn through port 14 and led through the classifier 15 to the recirculating system. Finished products having the desired particle size are withdrawn at port 16 in the classifier 15 and the liquid metal carbonyl bearing fine particles is withdrawn from the classifier through the port 17. Initial starting powders which may be coated in the apparatus may be fed through feeder 18, having hopper means 32 and means for feeding pressurized carbon monoxide 33 associated therewith, into the recirculating system and the make-up liquid carbonyl is introduced into the circulating system at 19. A pump 20, which may be a single stage or a multi-stage pump, raises the pressure of the liquid material fed thereto. It will be appreciated that pump 20, in combination with the throt-

ling device 21, insures that the pressure within the circulating system therebetween will be at a higher level than the pressure within reactor 11. Pressurized liquid carbonyl is led from pump 20 through pipe 22 into heat exchanger 23 where the pressurized liquid is raised in temperature while avoiding boiling of the pressurized liquid in the recirculating system by means of heating fluid, which may be steam, heated diphenyl or other suitable liquid heat exchange medium, introduced into the heat exchanger at 24 and exhausted therefrom at 25. The heated and further pressurized liquid carbonyl is led from the heat exchanger through pipe 26 through throttling device 21 located advantageously at the end of pipe 26 and adjacent the wall of reactor 11 back into reactor 11. The physical motion imparted to the further pressurized liquid carbonyl being ejected through the throttling device, which may, for example, be a valve, orifice, venturi tube, etc., together with the accompanying decrease in pressure causes boiling and rapid decomposition of a portion of the liquid carbonyl in reactor 11. Sufficient heat is imparted to the particulate material suspended in the liquid metal carbonyl and to the liquid metal carbonyl in the heat exchanger to supply the heat requirements for the decomposition reaction, including the heat of vaporization absorbed in vaporizing metal carbonyl in the decomposer. The energy level of the particle-loaded liquid carbonyl is elevated due to heating in the heat exchanger and additional energy for carrying out the decomposition reaction is imparted by injection of pressurized liquid carbonyl from the heat exchanger into the decomposer. If desired, additional heat may be supplied to the reactor 11 by means of internal and/or external heating coils, although such expedients may lead to plating problems and are less advantageous. Metal resulting from the decomposition plates upon particles of material in the vicinity and, in addition, new metal nuclei are formed. The carbon monoxide formed by the decomposition rises within the reactor, is led through reflux condenser 27 to condense at least a substantial portion of the metal carbonyl vapor accompanying the escaping carbon monoxide, and is led through offtake 28. Condenser 27 may be provided with cooling water inlet and exit ports 29 and 30. In order to maximize condensation of the metal carbonyl included with the carbon monoxide gas, it is advantageous to maintain the pressure within reactor 11 in the range of about 40 to 70 atmospheres. The carbon monoxide withdrawn at pressure is readily adapted for the formation of fresh metal carbonyl by reaction with metal, regardless of any metal carbonyl accompanying the carbon monoxide. Condensed liquid metal carbonyl from the reactor effluent may then be returned to reactor 11. It will be appreciated that for control of temperature conditions within the system, it is advantageous to provide insulation 31 about the equipment. It is advantageous to employ a series of pumps 20 such that the pressure increase increment in each pump is relatively low. In this way, the problem of plating within each pump is minimized. Suitable valves, purge points and other auxiliary equipment adapted for handling materials of the nature contemplated herein are also employed. It is to be understood that when the pressure-temperature conditions in heat exchanger 23 are relatively considerably higher than in reactor 11, smaller quantities of particle-loaded liquid metal carbonyl need be circulated than in cases where the pressure-temperature differential between heat exchanger 23 and reactor 11 are relatively low. Actual rates of circulation are maintained at a level sufficient to maintain solid particles suspended in the liquid metal carbonyl.

As noted hereinbefore, the method as carried out in accordance with the foregoing description can be utilized for the decomposition of metal carbonyl to the corresponding metal and for the coating of metallic and non-metallic particles with the corresponding metal. Particulate materials in the particle size range from submicron sizes up to about 30 mesh, e.g., up to about 100 mesh or

up to about 325 mesh, can be coated in accordance with the invention. Particles within the foregoing size ranges are readily suspended and dispersed in liquid metal carbonyls and coarser particles, while they may be employed in certain instances, present reduced surface area in the decomposer. When a process and apparatus as described hereinbefore and depicted in the accompanying drawing are employed for the purpose of producing coated metallic or nonmetallic particles such as particles of alumina, thoria, zirconia, titania, silica, magnesia, graphite and the like, as well as finely divided metallic particles such as aluminum, titanium, magnesium, iron, copper, nickel, chromium and the like, are metered to the system by means of feeder 32, it is found that operation in accordance with the method as described hereinbefore for the production of coated powders provides a tightly adherent and substantially uniform coating about the individual particles. It is advantageous to employ stirrer 12 since the stirrer provides high shear forces between individual particles suspended in the liquid environment in reactor 11 and prevents settling of particulate material in the reactor. The high shearing forces prevent agglomeration of the powdered material and provide for substantially uniform plating of individual particles thereof. It will also be appreciated that by means of the present invention the tendency for fine particulate material to agglomerate is decreased and the particles are caused to behave in an individual manner.

The particle loading, which can be employed in liquid metal carbonyl while maintaining the particles in suspension, can vary widely depending upon the size of the particles, the density of the particles, design of the equipment, power provided for stirring, and upon many other practical factors. For example, in the case of nickel particles having an average subsieve size of about 4 microns, up to about 250 pounds of powder can be suspended in one cubic foot of liquid nickel carbonyl. It is advantageous from the standpoint of production rate to maintain a substantial particle loading in the liquid metal carbonyl. Thus, the presence of solid heated particles in the region or regions within the reactor wherein decomposition is induced facilitates the decomposition reaction and also favors the decomposition reaction as compared to the opposing tendency for vaporization of the liquid metal carbonyl. Accordingly, the particle loading is maintained at a level of, e.g., about 1 pound or 5 pounds or 10 pounds per cubic foot of metal carbonyl, although, in many instances, a particle loading of at least about 20 pounds or 30 pounds per cubic foot of metal carbonyl is more favorable.

As previously noted, liquid metal carbonyl such as nickel carbonyl or iron carbonyl can be caused to decompose to the respective metal by subjecting at least a portion of a body of the liquid metal carbonyl at or slightly below the boiling point at the ambient pressure thereof to a slight reduction in pressure. Mechanical means such as the stirrer 12 in the accompanying drawing can be employed to effect the necessary reduction in pressure which need be of only a very low order.

In order to give those skilled in the art a better understanding of the invention, the following illustrative examples are given:

EXAMPLE I

Approximately 14 parts by weight of alumina having an average particle size of about 0.025 micron was placed in a mechanically stirred, externally heated high pressure autoclave. Liquid nickel carbonyl was added to the chamber and heated at about 190° F. for 40 hours while maintaining a pressure of about 50 p.s.i.g. The mechanical stirrer was operated at about 600 revolutions per minute (r.p.m.) during the entire run to keep the coated particles highly dispersed in the carbonyl bath. An overhead reflux condenser, operating at pressure, cooled with refrigerated water, was used to strip carbonyl

from the effluent carbon monoxide. About 725 parts by weight of coated material was produced analyzing 97.31% nickel, 0.65% carbon, and 2.04% alumina.

EXAMPLE II

Forty-five parts by weight of atomized aluminum having a particle size of about 13 ± 3 microns was coated with nickel, in a manner similar to Example I, by heating at 150° F. for about 24 hours while maintaining about 20 p.s.i.g. on the system. Approximately 120 parts by weight of coated material was produced. A good dispersion of the light aluminum powder was obtained together with a uniform coating of nickel on the aluminum particles.

EXAMPLE III

Fifty parts by weight of alumina having an average particle size of about 0.025 micron was treated as in Example I for approximately 50 hours at 190° F. and 50 p.s.i.g. About 300 parts by weight of coated material was produced analyzing 7.45% Al_2O_3 .

EXAMPLE IV

Two hundred ninety-seven parts by weight of coated material from Example III was further treated for 44 hours at 192° F. and 52 p.s.i.g. About 500 parts by weight of material analyzing about 3.68% Al_2O_3 was obtained.

EXAMPLE V

One hundred parts by weight of minus 325 mesh magnesium powder was treated as in Example I for 24 hours at an average temperature and pressure of 197° F. and 52 p.s.i.g. About 230 parts by weight of coated powder was produced, analyzing 41.04% magnesium, 58.82% nickel, 0.14% carbon and having a Fisher subsieve size of 4.72 microns. This run demonstrated that reactive magnesium powder can readily be coated with nickel in accordance with the invention. On the other hand, chemical precipitation processes employed in the art for the purpose of coating particulate materials are unsuccessful in the case of reactive powders such as magnesium. A portion of material produced in accordance with this example was blended with additional carbonyl nickel powder to provide a blend containing about 0.055% magnesium. The powder was then isostatically pressed at 70° F. and 30,000 p.s.i.g., was sintered at 2200° F. in a hydrogen atmosphere, was forged and rolled to strip about 0.005-inch thick in commercial equipment, including, successively, a steam forging hammer and cold rolls. The resulting strip contained 86% of the magnesium in an active metallic form.

EXAMPLE VI

One hundred parts by weight of Type A carbonyl nickel powder having a Fisher subsieve size of 4.72 microns was treated as in Example I for 21 hours at an average temperature and pressure of 193° F. and 50 p.s.i.g. About 140 parts by weight of coated powder was produced having a Fisher subsieve size of 6.07 microns. Microscopic examination of the coated material clearly showed that the characteristic thorns or hairs on the Type A powder had been rounded off, resulting in improved flow properties. A simple test was devised to illustrate the improved flowability of the coated powder. Equal portions of coated and uncoated material were fed into a 60° Pyrex funnel containing a one-half inch diameter orifice and checked for run-out time. Average run-out time for the coated powder was 12.5 seconds as compared to 45 seconds for the uncoated material. The coated powder produced according to this example was thus greatly improved for the purpose of direct powder rolling to forms such as strip.

EXAMPLE VII

Twenty-five parts by weight of 0.6 to 0.8 micron thoria powder was treated as in Example I for 50 hours at an

average temperature and pressure of 190° F. and 48 p.s.i.g. About 250 parts by weight of coated powder was produced containing 9.35% thoria.

EXAMPLE VIII

Liquid nickel carbonyl containing 200 pounds of nickel powder having an average subsieve size of about 5 microns per cubic foot of nickel carbonyl is charged to the decomposer as illustrated in the accompanying drawing at a temperature of 311° F. and a pressure of about 350 p.s.i.g. A portion of the hot pressurized nickel carbonyl is withdrawn from the decomposer, elevated in pressure by means of a pump to a pressure of about 400 p.s.i.g., heated by means of a steam heated heat exchanger to a temperature of about 320° F., and reintroduced into the decomposer through an orifice. Decomposition of the nickel carbonyl to metal takes place at a rate of about 500 pounds of nickel per hour. Nickel powder having an average particle size of about 6 microns is continuously withdrawn from the system with the remaining finer metallic powder being recirculated. Carbon monoxide resulting from the decomposition reaction is withdrawn from the decomposer through a liquid-cooled overhead reflux condenser, and fed to a carbonylating reactor containing finely divided metallic nickel for the production of additional nickel carbonyl. Fresh nickel carbonyl is introduced into the recirculating system ahead of the pump at the rate of 1,450 pounds per hour.

As previously noted, an advantage of the invention resides in the fact that the invention makes possible the production of metal-coated powders, e.g., nickel-coated powders, at a high production rate, with good dispersion of the powder being coated, and with formation of a tight adherent coating of metal about the individual powder particles. The resulting metal-coated powders are highly advantageous for use in powder metallurgy. For example, a portion of nickel-coated alumina containing about 2.7 weight percent alumina was blended with a sufficient quantity of carbonyl nickel powder to provide an alumina content of about one volume percent. The mixture was isostatically pressed, sintered, and forged to bar. For comparison purposes, a mechanical mixture of the same carbonyl nickel powder with 4 volume percent of uncoated alumina powder was pressed, sintered, and forged to bar in the identical manner. Portions of the thus-produced metal were subjected to a stress-rupture test at 1800° F. and 2,000 pounds per square inch (p.s.i.). It was found that the bar produced from the initial material containing the nickel-coated alumina had a rupture life of 307 hours. In contrast thereto, the bar produced from the initial mechanical mixture containing the uncoated alumina powder had a rupture life of only 3 hours. It is believed that those skilled in the art will appreciate that many advantages accrue in powder metallurgy through the use of nickel-coated and iron-coated powders of various kinds. Thus, it is well recognized in the art that a major problem in producing alloy articles by powder metallurgy is the problem of obtaining good sintering of an initial powder mixture with low porosity in the sintered article. When the initial starting mixture contains particles of a refractory nature, the sintering problems become particularly acute. For example, a well recognized problem in the powder metallurgy art is the problem of producing chromium-containing alloy articles such as stainless steel articles, nickel-chromium alloy articles, and the like. The practical problem involved apparently pertains to the formation of oxide film on the surface of chromium powders. This problem is greatly mitigated by a coating of nickel about the powders. In the case wherein chromium powders prepared without an oxide coating are directly coated with nickel in accordance with the invention, the problem is obviated altogether.

It is to be appreciated that the particulate material referred to hereinbefore can comprise a very broad spec-

trum. Thus, materials can be coated which are solid at the operating temperatures involved, which do not dissolve in the liquid metal carbonyl employed and which do not oxidize the liquid metal carbonyl. These requirements exclude hydrocarbons which are soluble in liquid carbonyls and strong oxidizing agents such as iodine, etc.

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. Thus, apparatus as contemplated in accordance with the invention may take forms other than that illustrated in the accompanying drawing. For example, the circulating system, including a pump, heat exchanger and injection means, may be enclosed within the pressure vessel housing the decomposer chamber. In such a case, it is advantageous to insulate the heat exchanger and heat supply means from the liquid carbonyl in the decomposer chamber and expose heat exchange surface only to liquid material within the circulating system. In this manner, liquid metal carbonyl exposed to the heat exchange surfaces is at a higher pressure and temperature than the liquid in the decomposer area. Again, a plurality of injecting means can be employed for injecting pressurized liquid metal carbonyl into the decomposer in particular circumstances. Such modifications and variations are considered to be within the purview and scope of the invention and appended claims.

We claim:

1. The method for decomposing a metal carbonyl from the group consisting of iron carbonyl and nickel carbonyl which comprises establishing a body of liquid metal carbonyl in a decomposing zone, dispersing particulate material in said body, subjecting said liquid metal carbonyl in said decomposing zone to pressure-temperature conditions such that said liquid metal carbonyl does not exceed the boiling point thereof, circulating a portion of said liquid metal carbonyl containing said dispersed particulate material to a pressurizing and heating zone wherein the pressure and temperature of said circulated metal carbonyl are elevated as compared to the pressure and temperature conditions in said decomposing zone but such that the boiling conditions for said circulated metal carbonyl in said pressurizing and heating zone are not exceeded, and injecting further heated and further pressurized metal carbonyl from said pressurizing and heating zone into said decomposing zone while stirring the liquid metal carbonyl in said decomposing zone to cause decomposition of metal carbonyl in said decomposing zone.

2. The method for decomposing a metal carbonyl from the group consisting of iron carbonyl and nickel carbonyl which comprises establishing a body of liquid metal carbonyl in a decomposing zone, dispersing particulate material in said body, subjecting said liquid metal carbonyl in said decomposing zone to pressure-temperature conditions such that said liquid metal carbonyl does not exceed the boiling point, circulating a portion of said liquid metal carbonyl containing said dispersed particulate material to a pressurizing and heating zone wherein the pressure and temperature of said circulated metal carbonyl are elevated as compared to the pressure and temperature conditions in said decomposing zone but such that the boiling conditions for said circulated metal carbonyl in said pressurizing and heating zone are not exceeded, and injecting further heated and further pressurized metal carbonyl from said pressurizing and heating zone into said decomposing zone to cause decomposition of metal carbonyl in said decomposing zone.

3. The method for decomposing a metal carbonyl from the group consisting of nickel carbonyl and iron carbonyl which comprises establishing a body of liquid metal carbonyl having particulate material dispersed therethrough with said particulate material having a metal surface corresponding to the metal of said carbonyl, subjecting

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said body of liquid carbonyl to pressure-temperature conditions such that said body does not exceed the boiling point thereof, removing a portion of said liquid metal carbonyl containing said dispersed particulate material from said body, increasing the pressure upon said removed portion to a level higher than that in said body, heating said pressurized portion to a temperature higher than that of said body while avoiding boiling thereof, and injecting said heated, pressurized portion into said body to induce within said body decomposition of metal carbonyl.

4. The method for decomposing a metal carbonyl from the group consisting of nickel carbonyl and iron carbonyl which comprises establishing a body of liquid metal carbonyl having particulate material dispersed there-through, subjecting said body of liquid carbonyl to pressure-temperature conditions such that said body does not exceed the boiling point thereof, removing a portion of said liquid metal carbonyl bearing said dispersed material from said body, subjecting said removed portion to conditions of temperature and pressure higher than the pressure-temperature conditions within said body, and injecting the resulting heated portion into said body to induce within a portion of said body decomposition of said metal carbonyl.

5. The method for decomposing a metal carbonyl which comprises establishing a body of liquid metal carbonyl subjecting said body to conditions of temperature and pressure such that said body does not exceed the boiling point thereof, removing a portion of said liquid carbonyl from said body, subjecting said removed portion to conditions of temperature and pressure higher than the pressure-temperature conditions in said body, and injecting the resulting heated portion into said body to induce within said body decomposition of metal carbonyl.

6. The method for decomposing a metal carbonyl from the group consisting of nickel carbonyl and iron carbonyl

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which comprises establishing a body of liquid metal carbonyl having particulate material dispersed therethrough, subjecting said body to pressure-temperature conditions such that said body does not exceed the boiling point, and stirring said body to cause decomposition of said metal carbonyl.

7. The method for decomposing a metal carbonyl which comprises establishing a body of liquid metal carbonyl having particulate material dispersed therethrough, subjecting said body to conditions of temperature and pressure such that said body does not exceed the boiling point thereof, and inducing within a portion of said body a reduction in pressure to induce decomposition of said metal carbonyl in a liquid environment.

8. The method for decomposing a metal carbonyl which comprises subjecting a body of liquid metal carbonyl to pressure and temperature conditions such that said body does not exceed the boiling point thereof, and inducing within at least a portion of said body a reduction in pressure to induce decomposition of said metal carbonyl in a liquid environment.

9. The method according to claim 1 wherein metal from the decomposition of said metal carbonyl is deposited upon said particulate material.

10. The method according to claim 3 wherein metal from the decomposition of said metal carbonyl is deposited upon said particulate material.

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