April 26, 1960 T. J. WALSH ET AL 2,934,331

APPARATUS FOR MAKING A METAL SLURRY PRODUCT

Filed Dec. 22, 1955 2 Sheets-Sheet 1

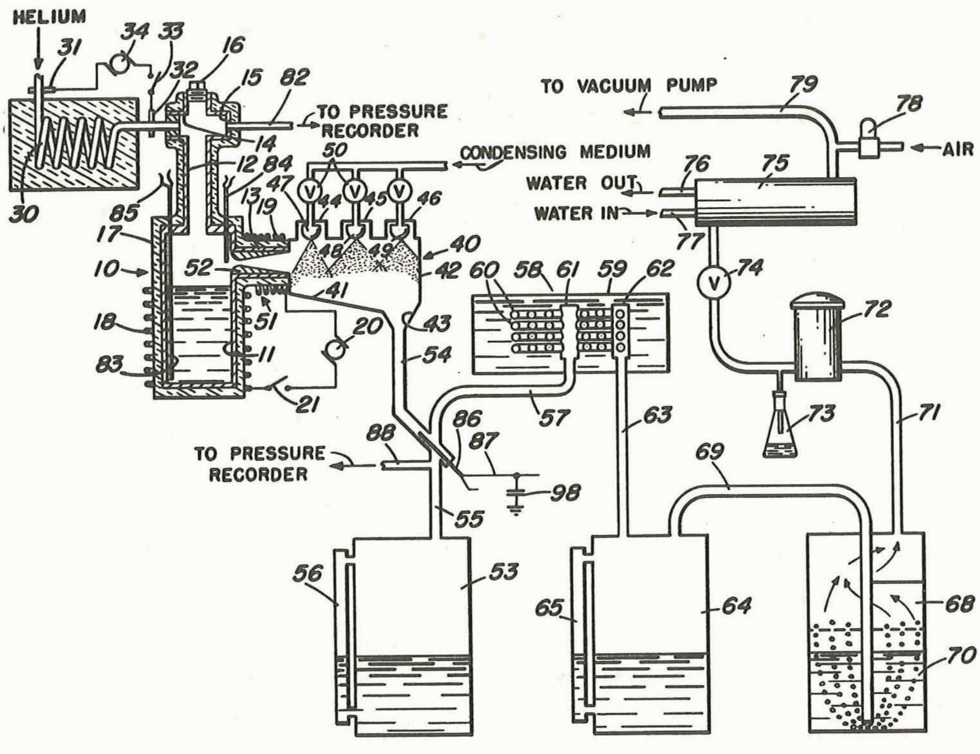

FIG. 1

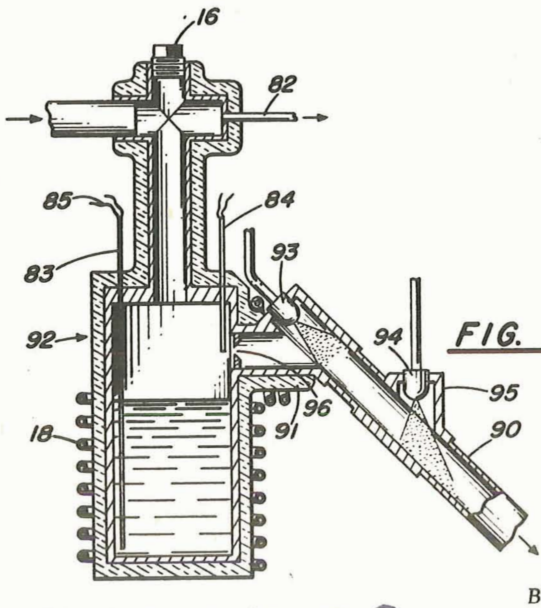

FIG. 2

INVENTORS
THOMAS J. WALSH
WALTER R. WITZKE
GEORGE M. PROK

BY

R. J. Tompkins

ATTORNEYS

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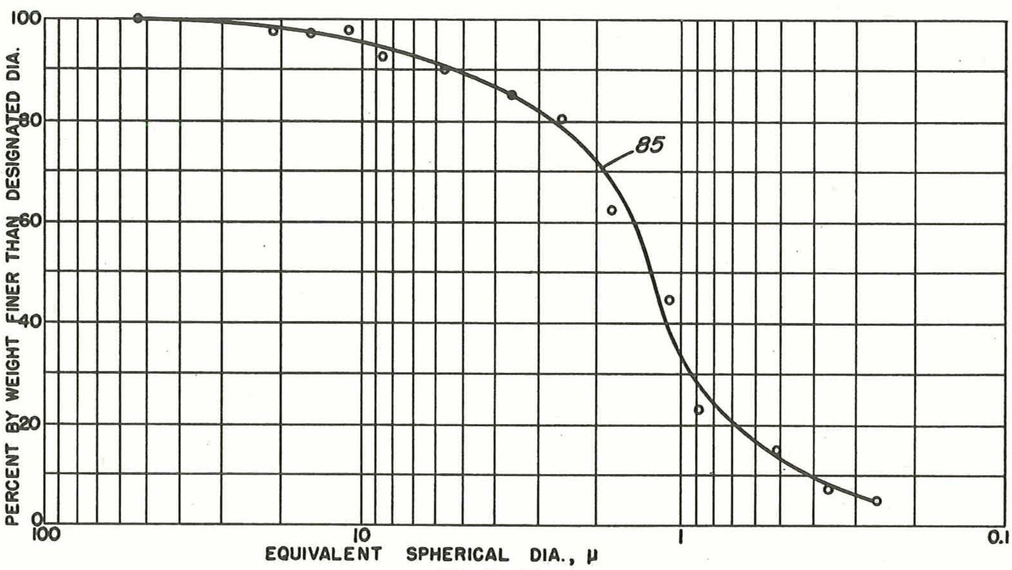

FIG. 3

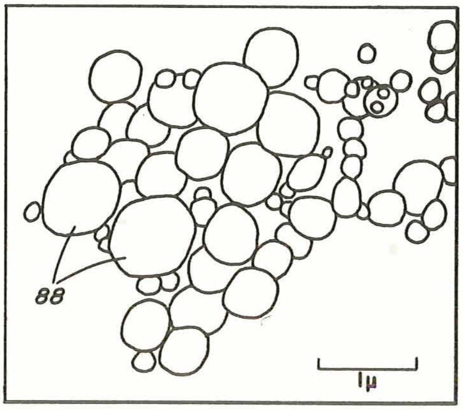

FIG. 4

INVENTORS
THOMAS J. WALSH
WALTER R. WITZKE
GEORGE M. PROK

BY F. J. Schmitt
R. J. Tompkins
ATTORNEYS

United States Patent Office

2,934,331

Patented Apr. 26, 1960

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2,934,331

APPARATUS FOR MAKING A METAL SLURRY PRODUCT

Thomas J. Walsh and Walter R. Witzke, Cleveland, and George M. Prok, Avon Lake, Ohio, assignors, by mesne assignments, to the United States of America as represented by the Administrator of the National Aeronautics and Space Administration

Application December 22, 1955, Serial No. 554,899

3 Claims. (Cl. 266—19)

(Granted under Title 35, U.S. Code (1952), sec. 266)

The invention described herein may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

This invention relates generally to the manufacture of extremely small metal particles especially in the form of metal-hydrocarbon slurries, and more particularly to apparatus for producing a hydrocarbon slurry containing extremely small particles of magnesium.

The preparation and use of metal slurries for various purposes are well known. However, the use of metal slurries for fuel in aircraft, particularly jet planes, has been attended with some important difficulties, such as improper flow and inadequate stability, arising largely from the oversize dimensions and improper shape of the metal particles, as produced by ordinary methods.

The objects of the invention include the provision of a process for preparing metal slurries in which the size of the metal particles is reduced to the upper limit of the colloidal range; the provision of a metal slurry-making process in which the use of mechanical disintegration process steps are eliminated; the provision of a metal vapor condensation process in which the coolant forms part of the slurry product; the provision of a metal slurry process in which clogging in the flow lines is largely eliminated; the provision of a metal slurry process in which impurities in the metal particles are effectively removed; the provision, in a metal slurry, of particles which have an average equivalent spherical diameter less than 2 microns and which have a purity ranging as high as 98.0% by weight free metal.

Other objects and many of the attendant advantages of this invention will be readily appreciated as the same becomes better understood by reference to the following detailed description when considered in connection with the accompanying drawings wherein:

Fig. 1 is a view, partly in section, showing the schematic arrangement of the apparatus used in the slurry making process;

Fig. 2 is a view of a modified vaporization pot;

Fig. 3 is a diagram of size distribution of the metal particles from a typical run; and

Fig. 4 is a copy of an electron micrograph of a representative field of magnesium particles obtained by the instant process.

Referring now more particularly to the drawings wherein like reference characters designate identical or similar parts throughout the several views, and more particularly to Fig. 1 thereof whereon apparatus for obtaining suitable metal slurries is shown as including a vaporizing furnace **10** consisting of an upright cylindrical casing **11** closed at the base and provided at the top with an inlet tube **12** and at the side adjacent the top with a lateral outlet tube **13**. The tube **12** terminates in a T structure having opposed side openings **14** and **15** and a screw-threaded end plug **16** which may be removed to

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provide access to the furnace interior. The entire furnace with the connecting tube and nozzle is covered with an asbestos insulation layer **17** and heat is supplied the furnace by induction coils **18** and **19** surrounding, respectively, both furnace and outlet tube. The coils are connected to a 30-kilowatt high-frequency electric source **20** through switch **21**.

Opening **14** of tube **12** is connected to a source of inert gas, such as helium, so as to provide, at will, a purging means for removal of oxygen from the entire unit and also, to permit operation of the unit under conditions of inert pressure, a value of 3.5 pounds per square inch being satisfactory flowing at a rate up to 23.7 pounds per hour. Without this pressure and, particularly, at sub-atmospheric pressures, the operation of the system results in particles having low free-metal content. This is assumed to be due to oxidation of the magnesium by air leaking into the apparatus, since on raising the pressure above atmospheric, high product purity is obtained. Preferably the helium is preheated prior to passage into the furnace by passage through an Inconel resistance coil **30** in series with the pipe line to the helium source. The coil **30** is provided with end terminals **31** and **32** which are connected through switch **33** to a 300 ampere direct current power supply **34**, such as used in welding equipment. A gas preheat in excess of the melting temperature of magnesium is required, that is, in excess of 1202° F., temperatures up to 1700° F. being usable, and temperatures below 1202° F. resulting in considerable, and in some cases, complete clogging of the flow line.

The outlet tube **13** of the furnace opens directly into the shock cooling condenser **40**. This condenser consists of an elongated chamber with a horizontal top and a base section inclined downwardly from the furnace forming larger and smaller ends, the smaller end **41** being attached to tube **13**, in extension thereof, and the larger end **42** being provided at its base with a funnel shaped outlet **43**. The top of the chamber is formed into a series of recesses, three as shown numbered **44**, **45** and **46**, respectively, in each of which is placed a single spray nozzle. These nozzles are of the hollow cone type, nozzle **47** preferably being rated at 10.5 gallons per hour with a 60° spray angle, and the other nozzles **48** and **49** preferably being rated at 21.5 gallons per hour with an 80° spray angle. Nozzle **47** with the narrowest spray angle is placed nearest the smaller end **41** of the condenser **40** to minimize the cooling of the furnace outlet tube **13** by the liquid spray. This condenser arrangement provides a curtain of coolant in the path of the hot metal laden vapor flowing through outlet tube **13** of vaporizing furnace **10**. Each spray nozzle is connected through a valve **50** to a source of condensing medium, which for fuel uses, should preferably be a hydrocarbon. Aircraft engine fuel, with an end point not over 300° C., a viscosity not exceeding 10.0 centistokes at −40° C. and a specific gravity not greater than 0.850 at 15.6/15.6° C. gives satisfactory performance.

The furnace outlet tube **13**, which feeds into the condenser **40** is preferably modified as a nozzle **51**, extending outwardly from the furnace chamber and provided with a contour insert **52** within the tube. This insert is made of stainless steel and is of the Venturi type with a rounded converging surface at the furnace end and uniformly diverging surface approaching the condenser end, the point of maximum constriction being near the furnace end.

To separate thet metal particles from the inert gas, the unvaporized hydrocarbon and part of the condensed magnesium particles are passed into a 20 gallon collector tank **53** positioned below the condenser **40** and connected to the outlet **43** of the condenser by tube sections **54** and **55**. This tank is provided with a depth gauge consisting of an external, transparent tube **56** connected to the tank at the top and bottom, as shown, to secure liquid depth measurement.

At the junction of tube sections 54 and 55 between the condenser and tank 53, a tube 57 leads to the cooling unit 58, which may be in the form as illustrated consisting of a flat shaped drum 59 adapted to be filled with ice-water, and containing cooling coils for condensation of the hydrocarbon vapors not condensed by condenser 40 or retained by the collector tank 53. These coils may take the form of helically wound layers of tubes 60 connected at their inner ends to a vertical manifold 61 in prolongation of tube 57, inside the drum. The outer ends of these coils are connected to manifold 62 and the manifold 62 feeds downwardly from the tank by tube 63 into a second collecting tank 64 also provided with a depth gauge 65.

As a further precaution in preventing loss of metal particles the inert gas is led from tank 64 to a point adjacent the base of a third tank 68 by means of tube 69, which enters tank 68 through the closed top thereof, as shown. This tank is about half filled with a hydrocarbon liquid 70 and the inert gas is made to bubble through this liquid to enforce particle deposition. The gas is then led by tube 71 through a felt filter 72, by a condensate receiver 73 through valve 74, through a water-cooled heat exchanger 75, provided with water connections 76 and 77, and by a bleed valve 78 to the vacuum pump line 79.

At appropriate points, measuring instruments, in addition to the liquid gauges of tanks 53 and 64, are employed to insure efficient operation of the unit. For example, at opening 15 of furnace inlet tube 12, a duct 82 connects the inflowing gas to a pressure recorder of conventional type, also thermocouple wells 83 for the liquid and 84 for the gas, are inserted in the furnace chamber and thermocouple connection 85 made to appropriate temperature reading instruments. A thermocouple well 86 and thermocouple elements 87 are placed, also, in the tube section 54 and a pressure recorder connection 88 made to the tube section 55, between the condenser and first collecting tank.

In operating the apparatus, the furnace chamber is first partly filled with magnesium metal. The system is then evacuated and purged with helium gas to remove all oxygen, and is then pressurized to about 3.5 pounds per square inch with this inert gas. With no flow of materials through the system, power is applied to heat the furnace, the helium and the nozzle 51. Sufficient heating of these three items is required to bring the temperature thereof above the condensation point of the furnace metal vapors so as to reduce the likelihood of condensation of these vapors between the furnace and shock-cooling condenser.

When the temperature of the liquid-magnesium in the furnace 10 reaches about 1400° F., flow of magnesium vapor is initiated in the flow circuit by reducing the pressure downstream of the furnace nozzle with the vacuum pump and starting the flow of helium gas through the gas heater 30 and through the furnace. At the same time the hydrocarbon is sprayed through nozzles 47, 48 and 49 into the condenser chamber. The pressure in the furnace, which tends to increase with temperature, is kept constant by means of manual adjustment of the helium flow; also, the pressure downstream of the nozzle is kept constant by manipulation of the bleeder valve 78.

To shut down the equipment the power to the induction heater of the furnace is cut off and the power to the helium heater is reduued. When the magnesium temperature decreases below 1200° F., the helium heater is turned off, the flow of helium reduced, and the vacuum pump and condensing liquid spray stopped, the magnesium cooling down to about 200° F. A slight positive pressure of helium is maintained throughout the cooling period.

The slurry recovered from the two collectors 53 and 64, the hydrocarbon bath tank 68 and the condensate receiver 73 are now pumped through a 100-mesh screen, not shown, to remove any coarse particles and thoroughly mixed with a turbine stirrer not shown, samples of the product being taken for analysis. While the slurry thus formed may be used without further treatment, it is usually desirable to concentrate the mix from a 0.5 percent solids value to about a 50 percent value, use being made of a bowl-type centrifuge, not shown, at a feed rate of about 500 cubic centimeters per minute to form an extremely viscous, clay-like mass which, when sliced, has a metallic luster similar to sodium metal. From this mass, by the addition of an appropriate surface-active agent such as 1.3 percent by weight of a glycerol sorbitan laurate, a pumpable slurry fuel is produced, this agent lowering the viscosity of the mass to between 5,000 and 10,000 centipoises.

The 100-mesh screen serves as a measure of the efficiency of the condensation process, this size screen being selected since the openings therein are smaller than those of any orifice which might be used in a fuel system and therefore precludes any future stoppage of slurry fuel flow. The amount of fine particles of magnesium passing through the screen ranged from 85.0 to 99.7 percent, on a weight basis, the average running close to 95.0 percent by weight. The particle-size distribution of the metal particles was determined by centrifugal sedimentation analysis, and for a typical run, was found to indicate that 33 percent by weight of the particles are finer than 1 micron in equivalent spherical diameter and 73 percent by weight finer than 2 microns. This relationship is shown in Fig. 3 of the drawings by curve 85.

The shape and size of the solid-metal particles obtained from the magnesium vapor condensation is best observed by means of electron micrographs of the product. Fig. 4, which is a copy of a micrograph enlarged 25,000 times, brings out the near colloidal size of the particles as well as their tendency toward a hexagonal shape.

The purity of the solid particles ranged as high as 98.9 percent by weight of free magnesium.

The various method steps and apparatus units as above described typify successful procedures used in obtaining the product. In lieu of these steps and units, other equivalent means may of course be made. For example, a hydrocarbon is specified as the condensing medium since the process is described for slurry fuel production. For other uses, other media are available, the main requirement being a boiling point sufficiently low to permit operation at the afterspray pressures in the system. A usable modification of the condenser, also, is illustrated in Fig. 2. In this arrangement the condenser takes the form of a straight tubular chamber 90, attached adjacent one end to the outlet tube 91 of furnace 92 and at the other end connected to the collecting system through tube section 54, as in the arrangement of Fig. 1. There are two spray nozzles in this chamber for coolant, one, 93, at the furnace end of the chamber 90 and directed axially therethrough, and the other, 94, supported vertically in an offset shell 95 above an opening in the chamber wall, so that metal-laden vapor is sprayed successively by down-stream directed coolant. In this arrangement, a short sharp-edged orifice 96 is used which has the smallest area at the furnace edge and sides expanding outwardly. In both condenser arrangements, the nozzle at the furnace outlet opens into enlarged condenser spaces so as to insure adequate and full gas flow with intimate mixing contact with the condensing mediums.

It is pointed out that using a high mass flow of heated helium gas through the outlet nozzle prevents the hydrocarbon from backing into the vaporizer and cracking; also, the hot helium, in conjunction with the heated outlet nozzle, tends to prevent the metal vapors from condensing and solidifying in the orifice. In order to eliminate alternating current disturbances from the induction coil 18 on the thermocouples, each thermocouple is grounded through a capacitor 98, as shown, for example, applied to thermocouple 87 of Fig. 1.

Obviously many modifications and variations of the present invention are possible in the light of the above teachings. It is therefore to be understood that within the scope of the appended claims the invention may be practiced otherwise than as specifically described.

What is claimed is:

1. Apparatus for preparing a slurry having minute metallic particles suspended therein comprising a furnace wherein the metallic material is vaporized, means coupled to said furnace for introducing an inert gas therein, a condenser coupled to said furnace for providing a liquid coolant spray in the path of travel of the vaporized metal flowing thereto from said furnace thereby to rapidly condense said vaporized metal into minute solid metallic particles suspended in said coolant, a first receptacle positioned below said condenser and coupled thereto for collecting the unvaporized coolant and the metallic particles suspended therein, means coupled to said condenser and to said first receptacle for condensing the coolant vapor, a second receptacle positioned below said last recited means for collecting the condensed coolant and the metallic particles suspended therein, means including a coolant bath and a felt filter coupled to said second receptacle for effecting separation of the metallic particles from the inert gas, and means including a vacuum pump upstream of said coolant bath for selectively controlling the flow through the apparatus.

2. Apparatus for preparing a hydrocarbon-slurry having minute metallic particles suspended therein comprising a furnace wherein the metallic material is heated and vaporized, means coupled to said furnace for introducing an inert gas therein at a rate necessary to maintain the pressure within said furnace at a desired level when the

temperature of the vaporized metal has reached a certain level, a shock-cooling condenser coupled to said furnace for providing a liquid hydrocarbon coolant in the path of travel of said vaporized metal flowing thereto from said furnace thereby to rapidly condense said vaporized metal into minute solid particles suspended in said coolant, a first enclosed receptacle positioned below said condenser and coupled thereto for collecting the unvaporized hydrocarbon coolant and the metallic particles suspended therein, means coupled to said condenser and to said first receptacle for condensing the vaporized hydrocarbon coolant, a second enclosed receptacle positioned below said last recited means for collecting the condensed hydrocarbon coolant and the metallic particles suspended therein, means including a hydrocarbon bath coupled to said second receptacle for effecting separation of the metallic particles from the inert gas, a heat exchanger coupled to said last recited means for condensing hydrocarbon vapors flowing therethrough, and means including a vacuum pump upstream of said heat exchanger for selectively controlling the flow through the apparatus.

3. Apparatus according to claim 2, and including means for pre-heating said inert gas.

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