To all whom it may concern:

Be it known that I, CARLTON ELLIS, a citizen of the United States, and a resident of Montclair, in the county of Essex and State of New Jersey, have invented certain new and useful Improvements in Apparatus for Making White Lead, of which the following is a specification.

This invention relates to a process of making white lead and similar pigments, and to apparatus therefor; and relates particularly to the process and apparatus for manufacturing white lead in a substantially continuous manner; all as more fully hereinafter described and as claimed.

The manufacture of white lead by the old Dutch process involves treatment of the metallic lead under corroding conditions for a space of two or three months, and on account of the protracted time required for the complete corrosion of the lead, together with the labor cost involved in handling the raw and finished product, the cost of manufacturing white lead by this process is relatively high. For this reason numerous processes have come into existence which involve the preliminary comminution of the metallic lead in some way, and subsequent treatment of the comminuted product with air, carbon dioxide and moisture; sometimes in the presence of acetic acid or other similar chemical reagents. The old Dutch process as well as the quicker methods of comminution usually require soft refined lead. Suitable corroder's lead generally commands an extra price and is expected to be free from such impurities as iron, copper, antimony and arsenic. If these impurities are present they constitute a source of much trouble and irregularity in the processes of corrosion. Copper and iron both tend to discolor the resulting pigment. Antimony has a peculiar inhibiting action on corrosion. In large proportion it may even check corrosion altogether. When little is found present in large amount in the corroded stock the troubles may often be traced to antimony. In the old Dutch process the uncorroded lead, buckled residues and the like, are largely removed by rolling and washing. In the quick processes of making white lead, depending on the corrosion of comminuted lead by the action of air, moisture and carbon dioxide, with perhaps acetic or another acid present in small amount as a carrier, the removal of the unchanged or blue lead presents some difficulties and in some cases may be wholly impracticable. Hence complete conversion of the lead would be desirable, or failing that, a method which produces the minimum amount of blue lead and in a form readily separable from the corroded lead. Sandy lead, which forms in the Dutch process when the temperature of the corroding pots becomes too high, is a crystalline neutral carbonate. It is difficult to grind, lacks body and settles to the bottom of the container when thinned for painting. Sandy lead appears in the product of the quick processes, at times, especially if the metallic lead has been poorly or irregularly comminuted and the subsequent process of corrosion, in consequence, forced to the extreme, in an endeavor to overcome the defects of comminution. Again, in the quick processes the hydration of the lead largely precedes the carbonation stage; and in fact in some cases it is probable that this separation of the two reactions is essential.

In the present invention hydration and carbonation, if desired, may be carried out substantially simultaneously, with a considerable saving in time, labor and apparatus. The present invention seeks also that organization which makes possible the use of hard lead, which leads to the substantial reduction or elimination of blue lead and sandy lead and by substantially continuous operation or non-cumulative handling of the material throughout the major portion of the several operations involved effects a reduction in the cost of manufacture of the product. In the handling of a material involving so great a tonnage as white lead, small reductions in manufacturing costs effect a considerable total saving.

The process herein set forth also involves an especially thorough and uniform comminution of the metallic lead, preferably with the instantaneous formation of a coating of a peculiar suboxide on the minute particles or filaments of the metal. The hydration of comminuted lead may lead to the formation of several different hydrates and the progress of carbonation as well as the character of the final product depends very largely on the nature of the initial hydration, which, other things being equal, is
controlled by the degree and uniformity of comminution, and the character of its surface coating of oxid, or absence of oxid, as the case may be. Simultaneous hydration and carbonation of irregularly comminuted lead usually leads to the formation of objectionable sand and lead and excessive amounts of blue lead.

The present application contains matter which is contained in Serial Nos. 570,244 and 641,306.

The manner in which the present invention is carried out will be clear from the accompanying diagrammatic drawings, (not to scale) wherein—

Figure 1 shows the assembled apparatus in section and elevation; while Figs. 2 and 3 show certain nozzle types or metal atomizers in section. Fig. 4 shows a centrifugal comminuter in plan view. Fig. 5 depicts a plan view of a set of conveyors and Fig. 6, shows this construction in longitudinal elevation.

As stated, the drawings are purely diagrammatic, for the sake of simplicity of illustration, and are to be regarded as a preferred or illustrative embodiment wherein various changes or modifications may be made without departing from the spirit of the invention.

In the drawings, 1 is a melting pot having the grates 2, and comminuting nozzle 3. A pipe 4 extends down through the pot and terminates in the nozzle 3. The latter is fitted with a conical disintegrator 5. Beneath the pot is placed a receptacle 6 having an outlet 7, leading into the conveyor trough 8. The interior of this trough is shown by the exposed portion or section at 9, where propelling blades are seen mounted on a horizontal shaft. The conveyor and its various parts should be constructed of material which has no discoloring action on the product. In order to afford proper time contact the conveyor is of very considerable length. A jacket 10 is provided, into which steam may be admitted through the pipe 11; an outlet being provided at 12. Or 12 may be used as the inlet and 11 the outlet according to circumstances. When cooling is required, cold water may be admitted into this jacket to control the reaction. Inlets, 22, 23 and 24 and also 25, 26 and 27 are provided for the introduction of air, moisture and carbon dioxide. An outlet 13 leads to a separator 14 which by passage 16 communicates with the conveyor 17. The separator 14 also has an outlet 15. The conveyor 17 is arranged in a manner similar to the conveyor 8. The discharge outlet is shown at 21.

18 is a heating or cooling jacket supplied with a heating or cooling medium by the inlet pipe 19 and outlet pipe 20.

In Fig. 2, 3 is an outer casing tapered at one end 28 to form a nozzle opening 29. Metal ducts 32 and 33 terminate within this casing to form tips or metal nozzles angularly disposed with respect to each other, as shown by 30 and 31. An air or steam pipe 35 terminates in the nozzle 33' adjacent to the metal tips 30 and 31.

In Fig. 5 an annular chamber 34 is supplied with air, steam and the like by the inlet pipes 37 and 38. 36 is the metal supply pipe. 39 and 40 are conical disintegrators placed in front of the casing 3, in operative relation to the tip or nozzle thereof.

In Fig. 4 a rotatable drum 41 contains the molten metal, which is discharged through the outlets 42. 43 is an annular slot through which air, steam and the like are discharged against the issuing streams of molten metal.

The operation of the apparatus under the present invention is as follows—Molten lead or any suitable lead alloy fills the melting pot 1. The metal preferably should be in a supermolten condition, or at a temperature some 400 or 500 degrees above the melting point. The column of metal preferably should be high so as to furnish a very great pressure or head at the nozzle 3. The metal is allowed to pass slowly into the comminuting nozzle 3 and air or steam under high pressure introduced by the pipe 4. Communion of the metal takes place and as the stream passes through the conical comminuter 5 further reduction occurs, due in part to vortex action and wire-drawing of the air or steam current. The finely divided metal is collected in the chamber 6 and is discharged from thence in a regula manner into the converting conveyor 8. It is passed slowly along the conveying chamber and is subjected to the action of moisture and oxygen or air to accomplish or begin the reaction of hydration. In the case of a hard lead steam may be admitted into the jacket 10 to hasten the reaction. Very reactive leads may require cooling, in which case cold water is admitted into the jacket in lieu of steam. Carbon dioxide may also be introduced into the conveying chamber in order to secure partial carbonation. The material is at length discharged from the first conveyor or conveyor system and passes into the separator 14 wherein is removed from undesirable metallic particles and thus purified it passes into the carbonating chamber 17 where, in contact with moisture and carbon dioxide, and air if necessary, final conversion takes place and the product is discharged at 21. After dewatering the product is ready to be packaged for shipment or ground in oil. The comminuting nozzles herein shown are depicted in several forms, especially to emphasize the necessity of careful and uniform comminution of the metal to an excessively fine state. The importance of length of travel of the hydrating and carbonate elements...
bonating mass is also emphasized. The time contact factor may be increased without unduly increasing the length of the conveying system by the cyclic or shunt treatment shown in Figs. 5 and 6. In Fig. 5, which is a plan view of a set of conveyors, 44 represents the feed end and 46 the discharge end. 45, 47, 48 and 49 indicate conveyors operating in a manner to propel the material as indicated by the arrows. In Fig. 6, which is a longitudinal elevation of Fig. 5, the conveyor 46 is shown as somewhat elevated above the bottom of the other conveyors, thus forming the dam 50. One or more of these cyclic or shunt conveyors may be placed in the conveying line of the hydrating and carbonating apparatus above set forth. The operation is as follows,—material traveling along the conveyor line 44 and 45 reaches the dam 50, where the lighter portion of the material passes on by way of the conveyor 46. The heavier portion is caught by the conveyor 47, delivered to conveyor 48 and from thence by conveyor 49 reaches the feed end 44, thus making a complete cycle or shunt return. Meanwhile hydration or hydration and carbonation are in progress as the material is being constantly subjected to corroding influences;—air, water, steam, acetic acid, carbonic acid and the like, so that on arrival again at the dam 50 more conversion has occurred and the lighter products pass away by the conveyor 46. Fresh material is supplied constantly at the feed end 44 to make up the loss by withdrawal of the material in an advanced stage of corrosion at 46.

Ordinarily it is not necessary to make use of acetic acid or other strong chemical agent as air, moisture and carbon dioxide together reacting on the comminuted lead of a substantially uniform degree of extreme fineness accomplishes this result effectively and produces a pigment of especially high covering power. The use of acids or other reagents is not however debarred in the present process. Instead of air, pure oxygen or oxygen and nitrogen mixtures having the former component in larger proportion than is present in air, may be used. A high oxygen content leads to more rapid oxidation and hydration. The influence of temperature also is marked, slight increases in the temperature showing an immediate response in the rate of hydration.

The effect of using a high concentration of carbon dioxide, especially in the later stages of carbonation is also noticeable. Lime kiln gases, carefully washed, carrying 30% to 40% of carbon dioxide are useful. Pure carbon dioxide also may be used. A mixture of oxygen and carbon dioxide, without any diluting gas, may be used for forced operation.

As stated, the degree of fineness of the filamentous or comminuted lead is a most important consideration. Excessive comminution to a degree double or triple that heretofore practised in the art is one of the first considerations leading to a high grade product of pronounced covering power. So far as I am advised, ordinary impingement of a jet of air or steam and the like has not been carried out in a manner affording a product of the requisite degree of fineness to make possible the substantially continuous hydration and carbonation of a traveling stream, yielding a product of a satisfactory amorphous and free-working qualities. This defect of imperfect comminution may be in part due to the cooling effect of expansion of the compressed fluid jet.

For example, superheated steam under high pressure, suddenly ejected through an orifice and expanded to atmospheric pressure, falls in temperature sometimes many hundred degrees through absorption of heat due to expansion. Hence when the steam contacts with the molten metal it exerts a chilling action which causes coarse granulation. In the present invention the nozzle shown in Fig. 2 obviates this difficulty as the metal is initially comminuted by the mutual impingement of the molten streams, with almost simultaneous disintegrating action of the high pressure jet and the vortex action and wire drawing occurring in the conical disintegrator.

What I claim is:

1. An apparatus for making white lead comprising a receptacle for holding molten lead, adjacent thereto a metal atomizer adapted to comminute lead to a product of extreme fineness, a conveyer of great length adapted to propel the comminuted product, a shunt return forming a part of said conveyer, means for contacting said product while in said conveyer with air in the presence of moisture; whereby said product is hydrated; a separator connected with said conveyer adapted to free the hydrated material from substantially all metallic particles, a second conveyer adapted to receive and propel the hydrated material from said separator, and means for introducing carbon dioxide into said second conveyer; whereby the hydrated material is carbonated.

2. An apparatus for making white lead comprising a receptacle for holding molten lead, adjacent thereto a metal atomizer adapted to comminute lead to a product of extreme fineness, a conveyer of great length adapted to propel the comminuted product, means for contacting said product while in said conveyer with air in the presence of moisture; whereby said product is hydrated; a separator connected with said conveyer adapted to free the hydrated material from substantially all metallic particles, a second conveyer adapted to receive and propel the hydrated material from said separator, and means for introducing carbon dioxide into said second conveyer; whereby the hydrated material is carbonated.
rator, and means for introducing carbon
dioxid into said second conveyer; whereby
the hydrated material is carbonated.

3. Apparatus for making white lead com-
prising a receptacle for holding molten lead,
adjacent thereto a metal atomizer adapted
to comminute molten lead to a finely divided
product, a conveyer adapted to propel the
commuted product in contact with mois-
ture and with oxygenating and carbonating
gases, and interposed in said conveyer a
separator adapted to remove metallic par-
ticles from the material.

Signed at Montclair in the county of
Essex and State of New Jersey this 16th 15
day of August A. D. 1911.

CARLETON ELLIS.

Witnesses:

NATHANIEL L. FOSTER,
CHARLES WRIGHT.

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Washington, D.C."