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⑪ Publication number:

**0 497 074 A1**

⑫

## EUROPEAN PATENT APPLICATION

⑬ Application number: **91500011.1**

⑮ Int. Cl. 5: **B22F 9/08**

⑭ Date of filing: **31.01.91**

⑯ Date of publication of application:  
**05.08.92 Bulletin 92/32**

⑰ Designated Contracting States:  
**AT BE CH DE DK ES FR GB GR IT LI LU NL SE**

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㉒ **Industrial process to obtain powdered zinc.**

㉓ The invention is referred to an industrial process to manufacture zinc powder using nitrogen as a fractioning mean, in an injection between 10 and 40 atmospheres into a duct where the zinc drops by overflow at a temperature kept just below the boiling temperature, with the contribution of an oxidant gas measured to produce a 5% of surface oxidation, fractioning the mixture zinc-nitrogen with tangential or radial nozzles, keeping the zinc temperature and afterwards cooling it with nitrogen at low temperature and pressure, in order to extract finally the zinc powder and to recycle the nitrogen which is cooled, re-delivered, filtrated, and re-compressed and re-heated in a continuous cycle and a closed-circuit.

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## PURPOSE OF THE INVENTION

The purpose of the invention is a process to manufacture fine powdered zinc to be used in the industry characterized in that an inert gas is used as an atomizer gas, with a continuous recycling, making the most of the energy without any aggression to the environment.

## BACKGROUND OF THE INVENTION

Zinc powder is usually used in the chemical industry as a reducing agent, as a catalyst, and in the antirust paint manufacture. Its consumption in the EEC is continuously increasing, being actually estimated in eight to ten tons monthly. Antirust or anticorrosive paints are traditionally manufactured in base of zinc oxide and zinc chromate. However, these paints are not useful should the surface has any scrape, abrasion or fissure that would be enough to raise the paint cover. On the contrary, paints with pure zinc don't suffer the same consequences because the zinc creates a concentric area with a positive ion acting against the rust and, consequently, the fissures will not let defenceless the surface since the chemical and the ion effects continue protecting it.

So, it is not surprising that the demand of the market results in a production increasing and in a development of the manufacturing process in order to find higher efficiency and a better shape and size quality.

## STATE OF THE ART

Fine powdered zinc manufacture processes are actually two:  
BY DISTILLATION, evaporating zinc or any zinc alloy in a retort, passing this gas to a chamber without oxygen and sharply condensing it in powder at 300 °C. Condensation at higher temperatures -at about 410 °C- will result liquid.

Zinc powder is collected in a continuous way or at intervals of time according to the condenser and furnace characteristics.

This process, known since early this century and still in use in advanced countries, has the following troubles: energy, manpower and equipment high costs; low production; contaminant slags and self-combustion risk. There is no bibliography related to it.

BY ATOMIZING, introducing molten zinc and air compressed at 12 atmospheres into an atomizer.

The powdered zinc manufactured in this way has irregular shape and size, a low commercial value, and it is only used to manufacture electrolytic zinc to precipitate harmful ions.

The trouble of this process is that it is carried out at low temperatures to avoid any ignition risk and consequently the molten zinc particles expanded into the air are cooled and solidified into particles of big size with irregular shape.

In the 50's, John Wiley & Sons, a New York publishing house, published a book called "NON-FERROUS PRODUCTION METALLURGY" by John L. Bray, being published in Spain in 1962 by Ediciones Interciencia calling it "Metalurgia Extractiva de los Metales No Ferreos". On page 153, there is a short paragraph and a diagram dedicated to the description of this process with compressed air. Recently, simulating tests starting from metals with very low melting points have been carried out, but their data -related to lead, bismuth or tin at a temperature of 71 °C- are not transferable to metals with higher melting points such as, for example, the zinc at 400 °C.

## DESCRIPTION OF THE INVENTION

The invention proposes a process starting from the atomizing principle, with an inert gas, specifically the nitrogen, and with the zinc kept at a temperature higher than the melting point. This results in a dispersion of Zn and N, with the liquid particles having spherical shape due to the effect of the surface tension.

The atomized is solidified in a second area at low pressure and at a temperature below the melting point. The nitrogen-zinc mixture is cooled in a third area at a handling temperature.

The nitrogen is separated from the powdered zinc and the cooled gas is urged into a turbine at a low pressure to drive it in part to the second and third areas and in part to the compressor and to the heater to be again delivered to the molten zinc dispersion area.

When it has been said that the nitrogen is cooled, that is to say that its temperature is dropped below the zinc melting point in such a way to be possible the solidification of the zinc dispersed in the zinc initial injection and being later cooled to a handling temperature.

When the recovered nitrogen is moved at low pressure and temperature, a part of it is filtrated and re-compressed to be again used in the initial cycle, in such a way that it could be said that the nitrogen is always circulating in a closed-circuit. By the other hand, since the process is carried out in a continuous and automatic facility, it has a high efficiency, minimum manpower costs, and there is not any aggression to the environment.

One of the most important advantages of the process of this invention is the starting material. The electrolytic zinc, for example, is produced in big quantities, avoiding in this way the troubles of

other processes, such as the distillation, that uses hard zinc, which is not so easy to supply, has a lower efficiency, and also avoiding the production of iron, copper or lead slags, harmful for the environment.

## DESCRIPTION OF AN EXAMPLE OF PREFERRED REALIZATION

A zinc ingot is introduced into a melting furnace -induction resistance furnace, graphite crucible furnace, etc-by a tackle operated by a constant advance reducing motor in order to obtain a constant increase of the molten zinc at 500 °C.

The constant increasing of the molten zinc produces an overflow of molten mass which is driven by gravity towards an adjustable temperature furnace, similar to the melting furnace, which increases the temperature just below the zinc boiling point (910). This furnace is also provided with overflow means to drive the molten zinc to a syphon keeping there the slags, keeping in this a perfect isolation from the environment.

The syphon is also provided with overflow means concentrically arranged around a nozzle delivering nitrogen at a pressure between 10 and 40 atmospheres and at a temperature similar to that of the melting product (600-800 °C); the said syphon is heat resistant and its ends are provided with three or more tangential or radial delivering nozzles that disperse and divide the liquid mass into little particles.

The atomizing container is provided with a plurality of low pressure radial injectors (50-100 m/m of water column) which solidify the atomized at a lower level, whilst in a third area, at the same temperature and pressure, the nitrogen is being continuously introduced, letting the zinc powder at a handling temperature.

It is important for the exit gases of the furnaces to abate their temperature to an exchanger in order to drive the nitrogen directly, or with termic assistance, to a temperature close to the zinc boiling point.

It is also important to indicate -being this fundamental for the development of the invention- that the nitrogen injection feeding the delivering nozzle receives an oxygen contribution or atmosphere air in order to produce a maximum of 5% of surface oxidation in the periphery of the particles to avoid the bond by contact of the zinc liquid particles.

It is also important to state that in the ducts of the overflow means there is an inert atmosphere, such as the nitrogen, in order to maintain the zinc product without any environmental aggression.

Once the zinc powder has been cooled, a collector, which chamber forms a frame with the

atomizer, receives the solid particles, dropping a part of them by an exit aperture while the other part is drawn by a cyclone that, by centrifugalization, separates the solid from the nitrogen, passing this nitrogen to a cooler and from there urging it again by a low pressure duct to a pipe that ramifies by a part towards a filter, from where, once cleaned, is compressed, afterwards heated and finally injected again, while by the other part it is driven, once cooled, to the second and third areas.

## DESCRIPTION OF THE DRAWINGS

The figure of the drawings shows an industrial facility destined to the manufacture of zinc according to the described example of preferred realization, noting that the example and the drawing have only an illustrative character in respect to the invention and consequently they do not limit their practical application.

According to the diagram, a zinc ingot 2 is introduced into the melting furnace 1 -graphite crucible furnace, electrical induction or resistance furnace. The furnace has molten zinc at 500 °C of temperature adding zinc nearly to its overflow; at this moment, in a constant and progressive way, the ingot 2 is introduced hung from a chain 3 operated by a tackle, a differential or any other operating device with remote control and provided with its own mechanical means, as for example a reducing motor.

As the ingot 2 is introduced into the furnace 1, the liquid (the molten zinc) overflows by the duct 4, which has inside an inert atmosphere, towards the adjusting temperature furnace 5 with similar characteristics as the furnace 1 but with its operating temperature higher than that of furnace because it is increased just below 910 °C, which is the boiling temperature of the zinc. The adjusting temperature furnace 5 is provided with a duct 6 of the overflow means with an inside inert atmosphere; the duct 6 which ends in a syphon 7 is provided with a chamber 8 for slag retention and with overflow means 9 for the pouring of the zinc by a central duct 10 where a pipe 11 delivers nitrogen coming from a heater 12 which received temperature from the gasses emitted by the furnaces. These gasses act in a heat exchanger that forms part of the condenser, and once these gasses are cold they are neutralized before their exit to the environment. All this part which is complementary to the process has not been shown in order to simplify the diagram and to make easier its understanding.

It is important to stand out that the duct 13, which ends in the pipe 11 of the heater 12 that receives nitrogen at pressure coming from the compressor 14, incorporates an injector 15 of air

or oxygene from a resource 16. This contribution of air or oxigene is fundamental for the development of the process as it will be stated.

At the opening of the central duct 10 there are delivering nozzles 17 which are radially or tangentially arranged and that execute a dispersion of the liquid mass into little particles.

The oxigene or atmospheric air provided into the duct 13 is regulated in order to producto in the zinc a maximum of 5% of oxidation, an oxidation that flows to the external part of the particle rusting it in its surface in such a way that the particles of zinc will not bond by contact.

The zinc dropping by the central duct of the overflow means has a temperature below the 910 °C and the nitrogen delivered by the pipe 11 has a similar temperature. Given that this temperature is higher than the melting temperature, the dividing of zinc forms particles that by means of surface tension adopts an spheryc shape. The temperature and the inyection pressure (10 to 40 atmospheres) is mantained in the inyectors 17 and the mass divided in a nitrogen atmosphere is cooled in a second and a third level by low pressure inyectors 18, for example between 50 and 150 mm of water column. The nozzles 18 inject nitrogen at a low temperature (the closest possible to the environment temperature) in such a way that the zinc powder is at a handling temperature.

The atomizing container or chamber 19 ends into a collector 20 with a separator 21 and with an exit by gravity 22. The lower part of the chamber 19, identified as 19-A must be considered as a solidification chamber of the zinc.

The heaviest zinc powder particles, about 30% of the total, drop by the exit aperture 22 and the rest -zinc plus nitrogen- is absorbed by a cyclon 23 which by a part expels the zinc powder by centrifugalitation through the exit 24 and by the other part delivers the nitrogen to a cooler 25 from where it is partially redelivered by a low pressure pipe 26 (100-300mm of water column) in part to the nozzles 18 and in part to a filter 27 from where it is taken by the compressor 14. The advantage of the invention is not based in the use of an inert gas but in the fact that the dividing is done at a limit temeprature just below the boiling temperature, and this principle is valid for any other metal or for any zinc alloy.

Another advantage of the invention is the partial oxidation of the divided particle, avoiding them to bond each to the other, being this a guaranty of the success of the process and the particle size between 1.5 and 5.5 microns.

And another advantage of the invention in the fast cooling of the particles once they are perfectly spheccically formed.

## Claims

1. A process to manufacture powdered zinc, that being of the atomized type and using an inert gas to impel and to divide, is characterized in that the zinc is kept during the fractionation at a limit temeprature below the boiling temperature and in the inert gas it is inyected an oxidant gas in controlled measurements to produce a 5% maximum surface oxidation in the powder particles, being the inert gas at a temperature the closest possible to the molten zinc and being cooled after the formation of the particles that acquire a spheryc shape due to the surface tension.
2. A procees claimed as the previous claim, characterized in that, at the exit of the duct where the zinc is urged with the nitrogen + oxidant, there are inyectors-fractioners of the zinc-nitrogen mixture with a temperature and pressure similar to those of the nitrogen urging the zinc, being this temperature close to the limit below the boiling temperature during the time of fractionating of the zinc, being the zinc powder formed by the inyection at low pressure cooled at a handling temperature with cold nitrogen in one or two steps.

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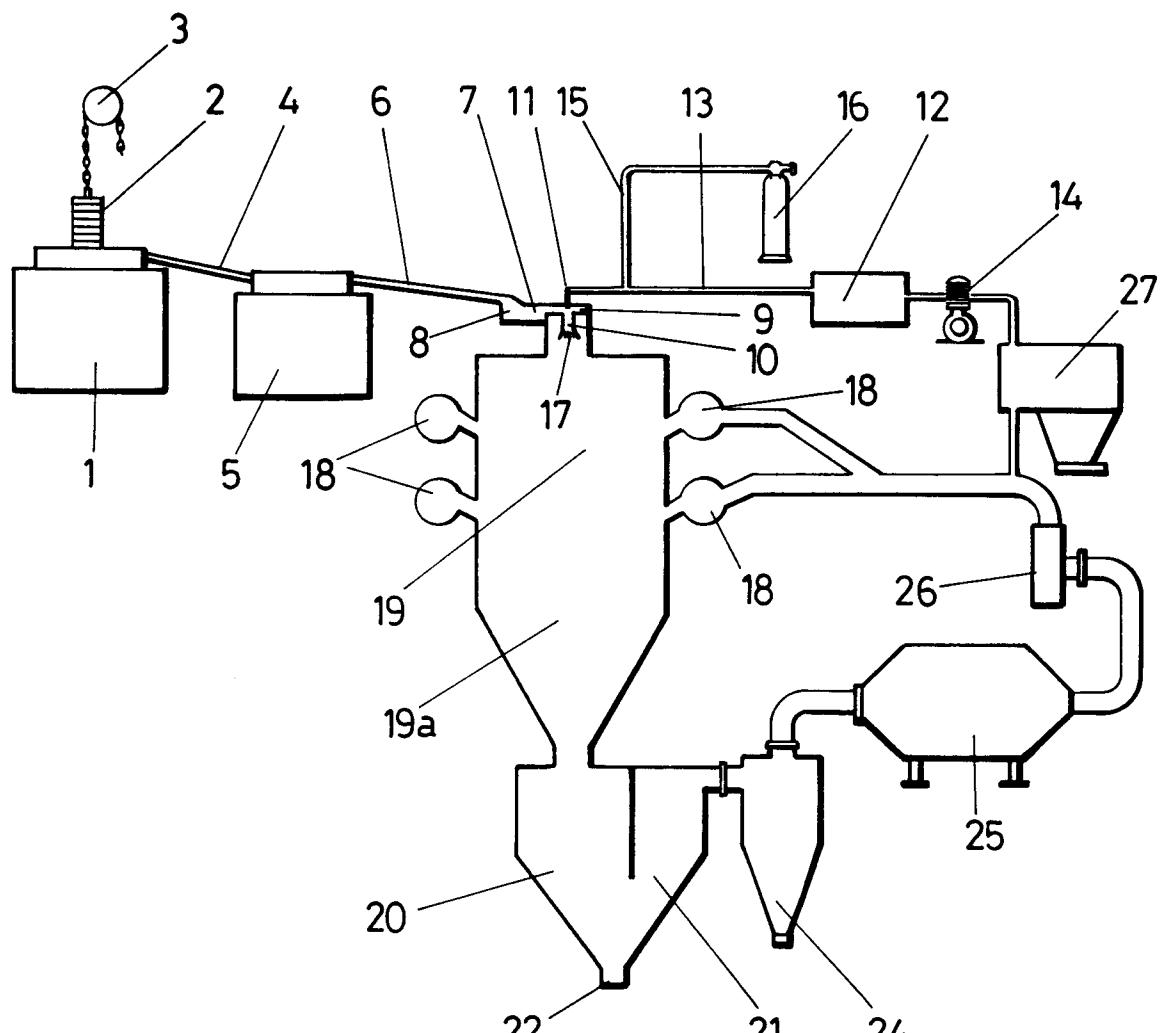


FIG. UNICA



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EUROPEAN SEARCH REPORT

Application Number

EP 91 50 0011

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
Y	GB-A-150 490 (J.P. MELLOR) * claims 1-2 * ----	1-2	B22F9/08
Y	PATENT ABSTRACTS OF JAPAN vol. 8, no. 69 (M-286) March 31, 1984 & JP-A-58 217 608 (TOYOU KINZOKUKU ) December 17, 1983 * the whole document * ----	1	
Y	GB-A-1 544 626 (BOC) * claims 1,4,16 * ----	2	
A	GB-A-1 364 899 (METALS&ALLOYS) * claims 15-18 * ----	1	
A	PATENT ABSTRACTS OF JAPAN vol. 7, no. 37 (M-193) February 15, 1983 & JP-A-57 188 603 (MATSUSHITA DENKI SANGYO ) November 19, 1982 * the whole document * -----	1	
TECHNICAL FIELDS SEARCHED (Int. Cl.5)			
B22F			
<p>The present search report has been drawn up for all claims</p>			
Place of search	Date of completion of the search		Examiner
THE HAGUE	19 SEPTEMBER 1991		SCHRUERS H.J.
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ..... & : member of the same patent family, corresponding document	
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			