

May 28, 1935.

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2,002,891

BRONZE, BRONZE POWDERS, AND METHOD OF MAKING THE SAME

Filed June 4, 1931

Fig. 1.

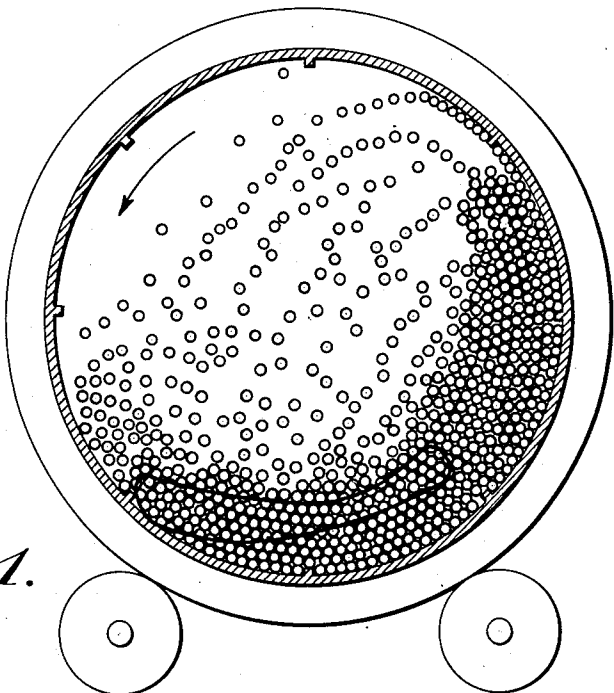
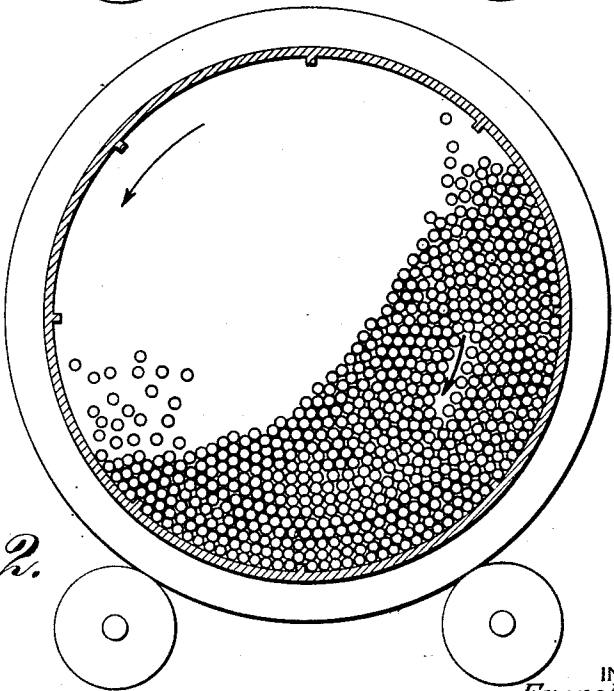


Fig. 2.



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2,002,891

BRONZE, BRONZE POWDERS, AND METHOD
OF MAKING THE SAME

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Application June 4, 1931, Serial No. 542,189

10 Claims. (Cl. 134—76)

This invention relates to the production of "bronze" powders, such as aluminum, copper and alloy "bronze" powder. Such powders are frag-
ments of metal in the form of flakes or plates or
scales. This flake form is desirable, in order that
a small weight of metal may be made to cover
the maximum surface and produce a metal-like
coating when applied as a paint. The flake form
of the particle is, however, only one important
feature of a desirable bronze powder. Another is
the requirement that when the powder is applied
as a paint, the flakes shall lie parallel to the sur-
face. How completely this result is attained de-
pends upon the characteristics of the flake sur-
face, and to some extent, upon the character of
the medium or vehicle in which the powder is
dispersed. Considering effective bronze powders
now known to the trade, it is believed that the
surfaces of the flakes are provided with a film
which gives the so-called "leafing" or "mirror-
ing" property and causes some of the particles to
float on the surface of the vehicle in the same
manner as a greasy needle will float on the sur-
face of water.

In current practice the metal in pieces of con-
venient size, to be converted to bronze powder,
is placed in a stamp mill with a small fraction of
a per cent of some lubricating material such as
tallow, olive oil, etc., and pounded or stamped.
As the particles are sufficiently reduced in size
they are either continuously or periodically re-
moved by screening or a current of gas. This
may entail several repeated operations. Follow-
ing the stamping and sizing operation the pow-
der is placed in a drum which is provided with
polishing brushes. These brushes are caused to
travel over the inner surface of the drum, giv-
ing a polishing action and cause to be distributed
over the surface of the particles stearic acid which
is added with the powder or subsequently. After
polishing for a number of hours varying with con-
ditions, the powder is then stored and allowed to
age. In this aging operation certain changes
take place which enhance the "leafing" property.

I have discovered that stearic acid or its equiv-
alent, when dissolved in a suitable liquid can be
made to impart expeditiously the desired "leaf-
ing" property. And, further, that this property
can be developed coincidentally with the grind-
ing or flaking operation. It has further devel-
oped that several factors are important in the
accomplishment of this result. For instance, if
the combined operation of flaking and film de-
velopment is to be carried out the grinding must
be by impact and not attrition, and the metal

must not be overworked. That is, there is an
optimum force or blow at which the metal par-
ticle should be struck and as the force increases
beyond that point results become progressively
less satisfactory. The temperature at which the
operation is carried on must be controlled, be-
cause here again an optimum condition exists.
There must also be a relation between the amount
of metal, the size of metal particle, the amount
of stearic acid and aluminum stearate, the amount
and kind of liquid, atmosphere and temperature.
These aforementioned factors are not capable of
calculation and therefore must be empirically ar-
rived at.

I have found that the leafing property can be
imparted to a clean flaked metal powder by treat-
ing it with the leafing agent in solution in a
suitable liquid, in the presence of an oxygen avail-
able atmosphere, and that the action is ac-
celerated by heating. As an example: A given
weight of flaked metal is heated with agitation
in the presence of air to 50° C. with five times its
weight of a 2% solution of stearic acid in benzene
and the excess liquid then removed. The result-
ing product will "leaf" in benzene.

As the leafing and flaking operations can be
more conveniently carried on simultaneously I
prefer to so operate. While the comminution can
be carried on in various grinding devices that
operate by impact I prefer to utilize a ball mill.

In the drawing, Figs. 1 and 2 illustrate a ball
mill such as may be used in the practice of my in-
vention.

Fig. 1 illustrates the proper operation of the
mill, as hereinafter set forth, so that the balls
act on the metal particles largely by impact; and

Fig. 2 shows what happens when the proper
operating conditions do not obtain, and there is
too much of attrition and rubbing action.

Per 1 pound of 100 mesh comminuted aluminum
(see paragraphs A and B) it has been found that
1 pound of a petroleum fraction oil, such as
"Varnolene" (see paragraph C), 3% of stearic
acid (see paragraphs D and E), 1% of aluminum
stearate (see paragraph E) and 20 pounds of balls
(see paragraphs F and J) give good results when
ground for seven hours (see paragraph G) in a
mill 16" diameter x 6" (see paragraph H) using
3/8" balls (see paragraph I) operating at 65
R. P. M. (see paragraph J) with a charge of
2 1/2 lbs. of aluminum (see paragraph K), at a
temperature of 50° C. (see paragraph L) and in
an air atmosphere (see paragraph D); or in a mill
24" diameter x 6", using 1/4" balls operating at
54 R. P. M. with a charge of 4.8 lbs. of aluminum.

A. Aluminum is used as an illustration because it is the bronze powder in greatest demand and most representative. The same principles, however, apply to other metals and alloys.

5 B. A 100-mesh is specified because it has been found convenient to disintegrate the metal to this degree of fineness before starting the flaking and leafing development operation.

10 C. A petroleum fraction such as "Varnolene" is selected as the grinding liquid because it is a common constituent of varnish, further, its boiling range is such that there is no excessive loss by evaporation or great fire hazard, yet the liquid can readily be removed by evaporation
15 when a dry product is desired. Liquids such as turpentine, benzol, or naphtha may be substituted for the "Varnolene", as the liquid in which the leaf or mirror-producing film is developed. It does not necessarily follow, however, that the
20 particles will leaf in this liquid. For instance, if the aluminum is ground in "Varnolene" it will leaf in that liquid, and also in the paint vehicle. If aluminum is ground in naphtha it will not leaf in the naphtha but will leaf in the paint vehicle.
25 The quantity used is adjusted to give the desired emulsion. (See paragraph E).

D. There is distinct evidence of chemical reaction between the metal and the stearic acid, in which oxygen plays a part. As grinding proceeds
30 the amount of stearic acid in solution decreases and a compound, probably aluminum stearate appears. If the grinding is carried on in a tightly closed mill there will be a drop in pressure, provided the temperature rise is not sufficient to give
35 a compensating vapor pressure. Further, if an oxygen-free gas, such as hydrogen, is used a drop in pressure does not occur and the leafing or mirroring action does not develop. The film-producing agent must have an affinity for the
40 metal being treated and develop a film which has a certain deportment with the vehicle of application, the exact nature of which is not definitely known. Addition of the agent may be made at beginning of the operation or toward the end.
45 While stearic acid is given as the film-producing agent, such substances as palmitic acid may be employed. Particularly where the leafing film is to be produced on the metal during the flaking operation, an excess of stearic acid in the sludge
50 as herein set forth seems to give a very good result. This may be for the reason that stearate dislodged from the flakes as by attrition during the milling operation does not re-attach itself, but the leafing film is repaired or restored by re-
55 action of some of the excess stearic acid with the denuded flake. 3% of stearic acid seems to provide a sufficient excess or reserve, though manifestly the proportion may be varied.

60 E. In the operation of a ball mill such as illustrated there are different zones of action (see J) and it is necessary to have the metal acted upon so far as possible in the impact zone. To prevent the metal from filtering through the interstitial spaces of the ball mass and appearing largely in
65 the attrition zone, it is necessary to produce an emulsion or froth carrying the metal particles which has sufficient body and tenacity to coat the balls and travel with them through the impact zone, yet permit sufficient mobility of the particles that they may present their flattened sides
70 to adjacent balls and mill sides on contact. Stearic acid and aluminum stearate are indicated for this purpose, though any number of substances may be employed, such as palm oil, oleic acid, olive
75 oil, palmitic acid, etc. The froth producing agent

may also have the function of imparting the leafing film to the metal during grinding, e. g., stearic or palmitic acid; or it may subserve only the froth producing function, e. g., palm oil, etc. In the latter case an additional agent will be employed to impart the leafing film, and the froth producing agent will be selected and/or controlled as to amount so as to hinder as little as possible the development of the leafing film.

10 F. A ratio of 20 to 1 of balls to aluminum has proven satisfactory for the efficient grinding with the production of a brilliant flake and good leafing quality.

G. The grinding time required is influenced by practically all the other factors mentioned and
15 fineness of product desired.

H. The length and diameter of mill determine its capacity and output and the selection of size is a matter of economics.

I. For the given set-up $\frac{3}{8}$ " balls were used, but
20 it is to be understood that the force required is dependent upon the physical properties of the metal and the size of the pieces being ground, and that this will influence either or both the ball size and the mill diameter. It is to be further under-
25 stood that the specific gravity of the metal being treated has a bearing on the ratio of metal to other factors. If it is desired to treat scrap foil or sheet directly they are first cut into pieces approxi-
30 mately $\frac{1}{8}$ to $\frac{1}{2}$ inch square and for foil 2/1000 to 4/1000 of an inch thick grinding can be carried on to advantage in two stages—first, $\frac{3}{4}$ " balls in a 16" mill and finished as above; second, for $\frac{1}{8}$ "
35 sheet 2 to 3" balls can be used to advantage for the initial stage.

J. The speed of a ball mill determines whether grinding is done by attrition (Fig. 2), or by impact largely (Fig. 1). When the mill is revolved the balls are carried up the side with a tendency to slippage. This slippage is retarded by the
40 insertion of internal ribs. When a certain height is reached, see Fig. 1, dependent on the charge, and below certain speeds, the balls in the upper layers roll down while the layers of balls adjacent to mill wall are being carried up with the balls
45 revolving on unoriented axes. The points in contact have a rubbing motion and therefore an abrading action which, in the lower layers, is intensified by the weight of superincumbent balls. This is not a satisfactory flaking action but results
50 in the production of an excessive amount of slimes, as well as being destructive of the leafing film. The effect is less pronounced with a decrease in the size of balls and an increase in the number of points of contact. By carrying the
55 proper charge of balls in the mill, about two-fifths full, and revolving at the proper speed the balls by a combination of centrifugal force and gravity, are carried up to a point where they are thrown clear of the mill shell and ascending balls
60 and fall in a free path striking the opposite side of the mill and balls that have preceded them, with the production of desired flakes. From the point of impact, in the direction of rotation, there is a zone of attrition but the major reduction in
65 particle size is by impact. For the most efficient grinding metal particles must lie between the impacting surfaces, and this is accomplished by the aforementioned froth or emulsion. It is also
70 patent that the size of the ball and diameter of the mill determine the force of impact. Too heavy a blow is destructive of the flake and the film surface and these two factors, with the resistance of the metal particles, determine the
75 ratio of mill diameter to ball size.

K. To properly charge a 16" diameter x 6" mill, 2½ lbs. of aluminum and 50 lbs. of balls are required for the desired height. Reducing the amount of metal increases the fineness of product, while conversely increasing the metal increases the coarseness of product, other things being the same.

L. 50° C. is adopted as a satisfactory temperature for operation. As the temperature rises above 50° C. there is a progressive tendency to loss of "leafing" and when at 100° C. it is seriously reduced.

The material as taken from the mill, with the removal of a part of the liquid, is a finished paste product without further treatment and can be stored indefinitely without losing its mirroring property. It is an established fact that ready mixed bronze powder paints lose their brilliancy on standing and therefore it is customary to market the powder and vehicle separately and mix on the job. This has several inconveniences due to the extremely light character of the powder. Therefore, I prefer to market my product in paste form and for that purpose an emulsifying agent such as aluminum stearate or tri-ethanolamine stearate is desirable. In grinding with stearic acid in solution there is apparently aluminum stearate formed and this formation increases with temperature and by proper control a satisfactory paste can be produced. However, I prefer to add aluminum stearate to the charge as it increases grinding efficiency as well.

A typical product in paste form contains 58% aluminum; 1% stearic acid; 1% aluminum stearate, which as stated helps the grinding action and also helps in the production and maintenance of the homogeneous paste form; and 40% of a petroleum fraction, such as "Varnolene". The flakes of the product possess the leafing quality, manifestable in ordinary paint liquids, and include no ingredients which will impair the leafing quality.

If a dry product is desired, the liquid can be evaporated under carefully controlled temperature and atmospheric conditions and the leafing property will be maintained. It is safe to heat the paste to 50° C. in an oxygen containing atmosphere; if the temperature goes much above there is a tendency to loss of leafing property, and, if heated until thoroughly dried, at 100° C., the leafing property is more or less completely destroyed in the presence of oxygen.

In a non-oxidizing atmosphere such as city gas it is permissible to dry the paste at 200° C. and above without destroying the leafing property. It is possible to use a vacuum in drying, but this lowers the permissible temperature as compared to drying in a gas atmosphere.

Because of the composition of the liquid, there will be no undesirable residue on evaporation. Thus if the product in its paste form should be accidentally subjected to evaporation there would be no dried paint film skin such as would make the product worthless as a paint pigment.

The principles of this disclosure have to do with bronze powders in general, while the details concern aluminum bronze powders, and it is not contended that they can be translated to other metals with equal results. As each metal has different properties, both physical and chemical, the factors must be adjusted to meet individual characteristics. It has been demonstrated, however, that a leaf-producing film can be developed by grinding under conditions as set forth on

aluminum, copper, zinc and tin, with no reason to doubt a similar behavior with their alloys.

Where particular materials are mentioned in the claims, it is to be understood that equivalent materials may be substituted, except where the language of a claim clearly excludes equivalents.

And where in a claim it is stated that a certain procedure, involving certain materials, temperatures, etc., is followed, substantially as set forth, reference is implied to typical examples given in the specification, not as limitative of the claim, but as illustrating the technique which constitutes the real invention,—empirically determined control and regulation of various factors, each in view of the other.

"Varnolene", referred to herein, as a petroleum fraction oil with a boiling point from in the neighborhood of 307° F. to around 405° F. (See U. S. patent to this applicant, No. 1,569,484.)

I claim:

1. The method which comprises imparting the quality of "leafing" or "mirroring" to flake metal powder by subjecting such powder to the action of a leafing agent dissolved in a volatile paint thinner, the amount of liquid being sufficient to form a sludge with the metal, and thereafter removing excess liquid to leave the residue in the form of a substantially homogeneous stable paste.

2. The method which comprises imparting the quality of "leafing" or "mirroring" to flake metal powder by subjecting such powder to the action of a leafing agent dissolved in a volatile paint thinner with oxygen available, the amount of liquid being sufficient to form a sludge with the metal, and thereafter removing excess liquid to leave the residue in the form of a substantially homogeneous stable paste.

3. As a new article of manufacture, a stable and homogeneous paste adapted for ready admixture with a paint vehicle, such as varnish, including flake bronze powder having on its surface a leafing film, the remainder of the paste comprising a paint thinner which is innocuous to the leafing film.

4. The method of producing bronze powder from metal particles, which comprises charging a ball mill with balls and with a quantity of the metal particles and a quantity of liquid containing in solution a leafing agent of the kind and for the purpose set forth, which imparts a leafing film to the metal and forms a creamy sludge with the metal in the operation of the mill, rotating the mill with oxygen available at such speed as to cause cataracting of the balls and to throw them clear of the mill shell and ascending balls, continuing the rotation until the metal has been brought to the desired fineness and flake form, maintaining in the mill a temperature favorable to the creation and maintenance of the leafing film, removing the sludge from the mill, and reducing the proportion of liquid to metal to such an extent as to produce a substantially homogeneous bronze powder paste.

5. A method which comprises imparting the quality of "leafing" or "mirroring" to flake aluminum powder by subjecting such powder to the action of a leafing agent dissolved in a volatile paint thinner, and in the presence of an oxygen containing atmosphere, forming a leafing film comprising aluminum stearate upon such particles and removing the liquid and preventing loss of the leafing film.

6. The method which comprises imparting the quality of "leafing" or "mirroring" to flake metal

powder by subjecting such powder to the action of a leafing agent dissolved in a volatile paint thinner, and in the presence of an oxygen containing atmosphere and removing the liquid in an oxidizing atmosphere at a temperature of substantially about 50° C., whereby loss of the leafing film is prevented.

7. The method which comprises imparting the quality of "leafing" or "mirroring" to flake metal powder by subjecting such powder to the action of a leafing agent dissolved in a volatile paint thinner, and in the presence of an oxygen containing atmosphere and removing the liquid in a non-oxidizing atmosphere at a temperature not substantially higher than about 200° C., whereby loss of the leafing film is prevented.

8. The method of producing bronze powder from aluminum particles, which comprises charging a ball mill with balls and with a quantity of the metal particles and a quantity of liquid containing in solution a leafing agent of the kind and for the purpose set forth, which imparts a leafing film to the metal and forms a creamy sludge with the metal in the operation of the mill, rotating the mill with oxygen available at such speed as to cause cataracting of the balls and to throw them clear of the mill shell and ascending balls, continuing the rotation until the metal has been brought to the desired fineness and flake form, maintaining in the mill a temperature favorable to the creation and maintenance

of the leafing film, removing the sludge from the mill, and separating liquid constituents therefrom; the solution comprising a liquid having the properties of "Varnolene" in which is dissolved stearic acid, the "Varnolene" being in the proportion of one pound per one pound of aluminum, and the stearic acid being in the proportion of 3% of the weight of aluminum.

9. As a new article of manufacture, a mixture of about 58% of flaked aluminum particles, about 1% stearic acid, about 1% aluminum stearate, and about 40% "Varnolene", the flaked aluminum having a leafing film, and the mixture being in a substantially homogeneous and persistent paste form.

10. As a new article of manufacture, a wet pigment for admixture with a paint vehicle for the purpose of forming a paint, comprising flaked metal bronze powder, the powder flakes having on their surface a leafing film as evidenced by the ability of the flakes to leaf in a paint vehicle, such as varnish, and being dispersed in a liquid carrier innocuous to the leafing film and which forms with the flakes a stable paste in which the flakes retain their color and leafing quality during storage, as evidenced by the fact that the paste can be heated at temperatures up to about 50° C. without substantial impairment in those respects.

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