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METHOD OF PRODUCING DISPERSION
STRENGTHENED METALS

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ABSTRACT OF THE DISCLOSURE

A method for dispersing refractory particles in a metal
having the steps of saturating the molten metal with anions
of the refracting particles, adding the refracting particles,
and agitating the molten metal.

This invention pertains to methods of producing dispersion
strengthened metals by adding the dispersoid to the molten
state of the metal thereby resulting in faster and more
economical strengthening of the metals and furthermore
extending the applications of dispersion strengthening.

In dispersion strengthening, a metal is formed with
small particles of a refractory material evenly dispersed
therein. These refractory materials may be oxides,
nitrides, carbides, silicides, or borides and in these
refractory materials the anions are respectively oxygen,
nitrogen, carbon, silicon, and boron.

In prior methods, the refractory particles were
homogeneously dispersed by several methods. In one
method, the metal was made into a fine powder and the
refractory particles were made into a fine powder, the
powders were mixed thoroughly; and the mixed powders
were compressed and then the powders were heated until
they were sintered. This method was expensive and time
consuming due to the necessity of making the metal into a
powder.

Another method was to put a metal salt in an
electrolytic bath, add the refractory particles, mix the
particles in the plating bath, and then the metal with the
refractory particles dispersed therein would plate out on the
electrode of the bath. This method again was expensive and
time consuming because of the low volume of metal that was
plated on the electrode.

A further prior method was to mix two alloys one
of which would contain the refractory particles and the
other of which would contain the metal in which the
particles were to be dispersed. With the proper mixtures
and temperatures, a precipitate would form which would
have the particles properly dispersed in the metal. This
again was time consuming and expensive.

It has been desired by the art for many years to be
able to add the refractory particles directly to the molten
metal to achieve the proper dispersion with resulting
strength and ductility improvements. This previously has
not been possible since when the refractory particles were
added directly to the molten metal, they would agglomerate
and would not achieve the degree of homogeneous
dispersion that was necessary for the desired metal
characteristics. This invention teaches a method whereby the
refractory particles may be added directly to the molten
metal without agglomeration. This is accomplished by
wetting the refractory particles completely with the metal,
whereby they will not agglomerate, and the wetting is
achieved by saturating the metal with the anion of the
refractory particle while the refractory particles are being
mixed in the molten metal.

There are many ways in which the molten metal can
be saturated with the anion, a few of which will now be
discussed. For illustrative purposes, an oxide refractory

particle will be assumed. The anion for an oxide particle
is oxygen and the metal would then be saturated with
oxygen throughout the mixing of the oxide refractory
particle in the metal. Such saturation can be achieved by
(1) supplying oxygen to the surface of the molten metal
at a partial pressure which is equal to or greater than
the equilibrium partial pressure at the solubility limit so
that no oxygen can leave the molten metal, (2) adding
feed stock to the molten metal which stock is high
enough in the concentration of oxygen so that the melt
is saturated with oxygen, (3) covering the melt with a
liquid containing oxygen such as a molten salt of oxygen,
(4) placing the melt in contact with sufficient material
to cause equilibrium to be reached and examples of this
would be the use of an oxide crucible or the covering
of the melt with oxide grain. These are examples of
manners in which the molten metal may be saturated with
the anion.

The time at which saturation of the molten metal with
the anion occurs may usually be determined by the time
at which slag formation on the surface of the melt occurs.
This slag formation should be kept to a minimum since
it may interfere with the mixing process. The slag may
be minimized by employing a reducing gas in the melt
to deoxidize the melt, again assuming the anion is oxygen.
The reduction should be controlled so that the melt is
not excessively reduced and the proper saturation
conditions are lost. Also, saturation may be determined
by periodically sampling the melt and analyzing the
sample for saturation. Slagging may also be controlled by
selecting a refractory particle which has an anion which
is slowly reactive to the metal.

As mentioned, the melt should be mixed in order to
obtain the proper distribution of the dispersoid. Preferably
the melt is mixed until the refractory particles are
uniformly dispersed in the melt to provide a substantially
homogeneous mixture. Such mixing may be achieved by
the use of a high energy propeller, a mixing screw, or
jets of inert gas. Mixing which produces cavitation has
been found satisfactory to adequately disperse the
refractory particles.

Satisfactory results have been obtained when the
refractory particles are in the size range between 100
angstrom units and one micron. In this range, sintering
and agglomerating of the refractory particles have been
prevented.

Table I, which follows, illustrates preferred refractory
particles for each of 17 different basis metals. The basis
metals may be pure or alloyed.

TABLE I

Basis metal	Refractory particles		
	Oxide	Carbide	Nitride
Tin	X	X	X
Lead	X	X	X
Zinc	X	X	X
Aluminum	X	X	X
Copper	X	X	X
Magnesium	X		X
Cobalt	X		X
Nickel	X		X
Iron	X		X
Chromium	X	X	X
Indium	X	X	X
Silver	X	X	X
Gold	X		X
Platinum	X		X
Molybdenum	X		X
Niobium	X		X
Zirconium	X		X

X—Indicates preferred combinations

A more complete list of refractory particles which may
be used in this invention is as follows:

Oxides of aluminum, silicon, titanium, zirconium, thorium,
uranium, yttrium, magnesium, beryllium, rare earth
oxides, and mixed oxides such as zirconates and spinels.

Carbides of titanium, zirconium, chromium, tantalum, tungsten, molybdenum, and silicon.

Nitrides of boron, silicon, hafnium, titanium, aluminum, and zirconium.

Silicides of molybdenum, yttrium, tantalum and zirconium.

Borides of chromium, zirconium, titanium, and tantalum.

In Table I, combinations that are indicated by an X are preferred for optimum strength and ductility. However, certain other combinations may be used for special purposes. As examples, several metals such as nickel, gold, and platinum dissolve a fair quantity of carbon in the melt which is later precipitated out as graphite. This normally adversely affects fabricability and strength so that these combinations do not exhibit the improved characteristics normally possible with this invention. However, these materials with the large quantity of graphite dissolved therein may prove useful as bearing metals or other special uses.

As will be appreciated by one skilled in the art, the more refractory and stable particles should be chosen for the higher melting metals and alloys.

Two specific examples of this invention will now be described. For the pure metal, 0.2 percent yield strengths are shown in the property tables of the following examples.

Example I

A hundred gram sample of lead with eight percent alumina of particle size 0.02 to 0.04 micron was melted and mixed with a propeller mixer in a Teflon crucible in air. The lead was melted and stirred until oxidation began to occur, and after a short period of time, the alumina was wet by the lead while being thoroughly mixed. The mixture was then briquetted, rolled and cut into samples for yield strength testing in compression, with the results given in the following table.

PROPERTY TABLE FOR EXAMPLE I

Temperature, ° F.....	Room	300	400	500
Lead, Example I p.s.i.....	6,000	4,500	2,500	2,500
Pure lead, p.s.i.....	1,200	0	0	0

The p.s.i. figures are yield strength in compression.

Example II

A hundred gram sample of tin with eight percent alumina of particle size 0.02 to 0.04 micron was melted and mixed with a propeller mixer in a Teflon crucible in an oxygen environment. The tin was melted and stirred until oxidation began to occur, and after a short period of time, the alumina was wet by the tin while being thoroughly mixed. The mixture was then briquetted, rolled, and cut into samples for yield strength testing in compression.

PROPERTY TABLE FOR EXAMPLE II

Temperature, ° F.....	Room	200	300	450
Tin, Example II, p.s.i.....	9,500	7,200	6,500	3,000
Pure tin, p.s.i.....	2,100	0	0	0

The p.s.i. figures are yield strength in compression.

Having thus described our invention, we claim:

1. The method of forming dispersion strengthened metals by dispersing refractory particles therein comprising the steps of

heating the metal until it is in a molten condition, substantially saturating the metal with the anion of the refractory particle to be dispersed therein, adding the refractory particles to be dispersed in the molten metal,

agitating the molten metal to sufficiently disperse the particles therein to obtain the desired characteristics.

2. The method of forming dispersion strengthened metals by dispersing refractory particles therein comprising the steps of

heating the metal until it is in a molten condition, substantially saturating the metal with the anion of the refractory particle to be dispersed therein,

adding the refractory particles to the molten metal,

agitating the molten metal to achieve substantially homogeneous dispersion of the particles therein.

3. A method of forming dispersion strengthened metals by dispersing refractory particles therein comprising the steps of

heating a metal having a component therein selected from a group consisting of tin, lead, zinc, aluminum, copper, magnesium, cobalt, nickel, iron, chromium, indium, silver, gold, platinum, molybdenum, niobium, and zirconium until it is in a molten condition,

substantially saturating the metal with an anion from the group consisting of oxygen, carbon, nitrogen, silicon, and boride, of the refractory particle to be dispersed therein,

adding the refractory particles to the molten metal, agitating the molten metal to obtain substantially homogeneous dispersion of the refractory particles.

4. The method of claim 1 having the step of preparing the refractory particles so that they have a diameter between 100 angstroms units and one micron.

5. The method of claim 1 wherein the step of saturating the metal with the anion of the refractory particle to be dispersed therein comprises supplying a partial pressure of the anion above the melt which is equal to or greater than the equilibrium partial pressure at the solubility limit of the anion in the metal.

6. The method of claim 1 wherein the step of saturating the metal with the anion of the refractory particle to be dispersed therein comprises adding to the molten metal sufficient quantities of metal stock having the anion therein to obtain saturation.

7. The method of claim 1 wherein the step of saturating the metal with the anion of the refractory particle to be dispersed comprises placing the molten metal in contact with material having the anion therein.

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