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**COMPOSITIONS AND ARTICLES INCLUDING
NON-PYROPHORIC MICROPARTICLES**Michael Walter Freeman, 401 David Whitney Bldg.,
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No Drawing. Continuation-in-part of application Ser. No. 225,901, Sept. 24, 1962, now Patent No. 3,276,921, dated Oct. 4, 1966. This application Oct. 22, 1965, Ser. No. 502,506

5 Claims. (Cl. 252—12)

This application is a continuation-in-part of prior application, Ser. No. 225,901, now Patent No. 3,276,921, filed Sept. 24, 1962, entitled, "Compositions and Articles Including Non-Pyrophoric Microparticles and Methods of Making and Using the Same."

This invention relates to composition and articles including metallic microparticles treated to be non-pyrophoric and methods of making and using the same. Because of the unique properties exhibited by such sub-micron size particles they may be used in and of themselves without admixture of other metals or non-metals but may be used desirably in admixture with other particles of larger size to which they will transmit unique properties carried over into articles made from such mixed compositions.

The metal powders set forth below are used in the present invention and are most desirably superfine powders of particle size of the order of particles of tobacco smoke, in that they are submicron in size, desirably of a range of from 0.01 to 0.1 micron, but sizes of about 0.01 to about 3 microns are useful for many purposes including particularly the range from about 0.05 to 1.0 micron.

While a wide variety of metal powders are included in the present invention most desirably they are solid metals of the transition element groups (e.g. whose inner electron shield is filled) and include manganese, chromium, cobalt, nickel, copper, iron, tungsten, molybdenum, zinc, tin and zirconium and mixtures thereof with one another or with larger particle powders of larger particle size heretofore available in the art, particularly macro size particles to which latter unique properties are communicated in such mixtures. Beryllium is also included.

The particle types are dendritic in their genesis, and each particle seems to be a single monolithic crystal. The term monolithic means massively single, solid and uniform crystal in microcrystalline state. New World Dictionary, 1958 Ed. The single difference in particle types seems to lie in the extent and quality of this dendriticity. These types are designated for convenience as (a) "discrete particles," useful in medicine and as very active catalysts; (b) "needles" or "rod-like particles," useful for magnets and magnetic tapes; and (c) "dendrites," useful for structural parts and the making of alloys with other metals and non-metals. These dendrites are subclassed as "weak dendrites" with thin branches; "strong dendrites" with thick branches; "filled in dendrites," where the branches are even thicker, and finally, there is a form of the iron referred to as "platelets," which are useful for making magnetic cores.

As illustrative, reference may be made to colloidal copper crystals in the form of an aggregate of tiny cubes about $\frac{1}{3}$ micron on a side plus a distribution of small copper particles about $\frac{1}{10}$ micron on a side. There may also be a needlelike or nodular rod. Generally a distribution of copper needles, rods, and particles is present. Such morphologically different forms of copper crystals have not been reported heretofore.

Plates for nickel-cadmium batteries were made from nickel superfine powder, of this invention. Ordinarily nickel plates are sintered at 1800° F.; with the nickel powder of this invention, sintering may be accomplished

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at 1100° F. and lower, resulting in a greater surface area and a greater energy output. In the few electron micrographs taken of this material there was many aggregates of extremely tiny particles probably 50 A. or less.

5 The crystalline architecture of the single particles has an important bearing on the ultimate physical properties and applications of the powder and this is well illustrated by the iron particles particularly of dendritic form. Thus the basic crystallite building unit in the iron particle is a rhombic dodecahedron. The dodecahedron crystal is
10 made up, in situ, of unit cubic cells each about 2.88 A. per side. The ultimate crystallites may be single 100 to 500 A. rhombic dodecahedrons.

The iron crystallites for example develop corner to corner along their diagonals to form a needle or rod
15 growing in the direction perpendicular to the (111) planes of the cube. They form a single monolithic crystal, the term having the meaning set forth above. Under certain conditions nodular rods are formed when small
20 crystallites begin to develop at the corners. Under other conditions these side-arms or secondaries develop even further, and a dendrite is formed. There are three secondaries at any node, separated by 120°, and all grow toward the growing tip at an angle of 70.5° to the direc-
25 tion of primary growth.

Another form of the alpha iron crystals has been referred to as a "platelet." Many of these hexagonal sheets, instead of appearing smooth and structureless, are den-
30 dritic in two dimensions, with a skeleton of growth along directions parallel to their six sides. These platelet crystals are identifiable as alpha iron by selected area diffraction. They may demonstrate the Kikuchi N pattern with Kikuchi lines for a platelet 80 A. (0.008 m μ) or thinner.

The surface area per unit mass (or specific surface) is
35 very large for metal crystals of this invention. It ranges from about 16 square meters to more than 20 square meters per gram of iron powder. This means that any process which affects the bulk of the material but whose rate depends on the exposed surface area will be
40 markedly increased as the particle size decreases. This has been shown in alloying, compacting, or coreacting the elemental powder of this invention with non-metallic materials such as nylon, butyl rubber, natural rubber, as
45 well as with different metals or combinations of any two or more of these materials. Its rate of oxidation is rapid and it is therefore highly pyrophoric. For active elements of sizes smaller than a micron—such as the iron powder here—the energy associated with the surface becomes
50 an important part of the total.

The finer the powder, the greater its chemical reactivity. This is an important factor in the physico-chemical reaction between the iron powder here and other metallic and non-metallic reacting matrices, such as copper, nylon,
55 rubbers as set forth above etc.

The microscopic dimensions of these particles make for great differences in their properties when compared with macroscopic, large particles of the same element. As a solid metal behaves very differently from the atoms of which it is composed, so these fine particles have characteristics which are very different from those of bulk materials. At the same time, of course, compacts of the particles have properties which are a function of the particles of which the compacts are composed. The particles of this invention in many cases measure less than 50 atoms
60 across, approaching in this case atomic dimensions.

While many of the properties of the superfine powders of the present invention are characteristics of the various metals, other properties, such as magnetic properties, may be those of only one, or a limited class of metals. Magnetic properties may be illustrated by iron powders here. The unusually good magnetic properties are directly asso-
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ciated with the shape and size of the individual particles. As an exemplary illustration of magnetic properties of iron samples of the present invention, the following results of an areal analysis of the particles of fine iron samples of the present invention, are summarized in Table I below.

TABLE I.—A REAL ANALYSIS OF ALPHA IRON PARTICLES

Sample	LCF					HCF		HCF LCF	(I) (μ)	(d) (μ)	(I) (d)	Relative Coercive Force (1033-1=100)
	FID	DEN	WD	PL	P	NR	N					
5401	24	17	156	0	0	317	77	2.0	0.36	0.09	4.0	163
1033-1	59	188	94	0	10	189	69	0.7	0.39	0.16	2.5	100
1042	94	46	64	0	0	182	60	1.2	0.74	0.23	3.2	82
1078	132	24	93	6	5	124	29	0.6	1.27	0.39	3.2	38
1431-1	75	4	11	112	78	55	7	0.2	0.91	0.28	3.2	24

NOTE.—Where L.C.F.=Lower Coercive Force; H.C.F.=Higher Coercive Force; F.I.D.=Filled, in Dendrites; DEN=Dendrites; W.D.=Weak Dendrites; PL=Platelets; P=Discrete Particles; N.R.=Nodular Rods; N=Needles.

Here it is apparent that in samples of higher coercive force the (LCF) class of particles are relatively less abundant than the (HCF) particles, and the agreement is direct, except for sample 1042 where particle size itself has entered to depress the coercive force. Sample 5401 is excellent in both aspects of morphology and has a high coercive force as well. In the areal analysis it should be emphasized that the results are quoted for the type of mounting and sample preparation employed and are only comparable from this viewpoint.

The following results illustrate certain properties of metals of the present invention concerning tensile strength and ductility after sintering, for some iron powders of the present invention.

TABLE II

Iron Powder Type	Sintered Density, gm./cc.	Tensile Strength, p.s.i.	Elongation Percent in 1"
Freeman #1	7.27	42,200	29
Freeman #13	7.22	80,000	4
Freeman #13A	7.89	116,000	3

Formulas 1, 13 and 13A refer respectively to pure iron of the present invention; such iron alloyed with 5% copper ordinary; and such iron alloyed with 15% copper. Attention is called to the unusual densities, tensile strength, and elongations exhibited by the powders of the invention.

Data on the density versus the compacting pressure for these fine iron crystals of the present invention is as follows. At 30 t.s.i., the green density is about 4.08 g./cc.; at 40 t.s.i., about 4.4 g./cc.; at 50, 4.6 g./cc.; and at 60 t.s.i., 4.78 g./cc.

The following example illustrates other properties.

EXAMPLE I

A xylol slurry of iron (of the present invention) and ordinary copper powder mixture (copper) was briquetted at 20 t.s.i. into a flanged cylinder measuring 0.432 "OD × .305" "ID × .731" Flange OD. After three hours of sintering at 2000° F., a density of 7.34 g./cc. was reported. Percent shrinkage of the body was 15 and that of the flange was 16.75. This is reported to indicate that a part of complex shape made of this powder may have substantially respective uniform shrinkage along its different thicknesses.

In general, uses for the powders of this invention are inspired by the extremely fine particle size, high specific surface, free energy and apparently high diffusion potentials. These powders may be utilized most profitably to permit material compositions and structures which are unavailable or of poor quality from other metals.

All powders of this invention may be safely and satisfactorily handled and compacted in room-temperature air

after they have been cooled to about -25° F. After the powder has been compacted, the pyrophoric properties are still present but may be rendered stable by sintering as for example at temperatures of about 1100° C. or 1300° C. or by exposing the material to dry ice and commercial nitrogen atmosphere in a dry-box at room-temperature,

once the result has been achieved, for example about one hour. The powder mixtures identified as Nos. 1, 13 and 13A, exhibited unusual densities and tensile strength when compared to commercial powders of similar composition and processing, but made with ordinary iron. The elongation of 30% of powder mixture No. 1, pressed and sintered to a density of 7.27 g./cc., is considerably higher than the elongation of commercial powders, whose maximum elongation is 11%.

In slip casting, this process utilizes fine particles as a raw material and can be used to produce shapes not ordinarily considered possible for powder metallurgy. Slip casting also utilizes particle size as a controlled variable and high sinterability as a necessity. Composition gradients in nuclear fuel elements are possible by this technique. In roll bonding, the excellent green bonding characteristic of the powders of this invention lends itself to this technique for iron and alloy mixtures. A viscous, resin bonder may be used so that the material need only be cured, not sintered. Parts may be fabricated by hot pressing or hot coining. The unusual fineness of the powders is admirably suited for establishing structures analogous to those of the sintered aluminum powder development. The iron-aluminum alloys offer valuable materials. Their oxidation resistance is excellent; their melting point would indicate that dispersed phase hardening may raise the temperature at which usefully used to for example 1400° or 1500° F. It has been indicated that additions of 5, 10 or 20% of these ultra-fine iron particles to commercial reduced or electrolytic grades of iron or of other metals, will greatly increase the sintered density and strength.

The metals and elements utilized in the form of superfine powders may be any of the ferrous and nonferrous powders and mixtures thereof such as iron, nickel, chromium, copper and alloys thereof and particularly powders of iron, nickel or alloys of nickel, chromium, etc. in particle size of from about 0.01 to 3 microns but more particularly of submicron size as from about 0.01 to 0.3 micron and up to 1 micron.

The metals may desirably be prepared by electrolytic processes and desirably under controlled conditions, the different types of resulting particles being capable of accurate prediction, each type being desirably held within narrow limits of size distribution. For these purposes the flow of current through an electrolyte in the electrolytic cell is periodically stopped after a time interval that retains the crystal form desired. pH and current density variables are maintained within preferred ranges. These variables, generally when coupled with concentration and composition of electrolyte, will determine particle size and nature of the powder produced. Other factors include flow rate of electrolyte through the electrolytic

cell, temperature, macroscopic geometry of the cell and the electrode material.

Removal of the metal deposit from the electrode is desirably carried out by periodic reversal of the current particularly using an electrolyte flowing through the cell. Such periodic reversal of current is an important factor in control of the structure of the powder. In general a period of about 20 to 300 or more desirably 30 to 130 seconds is desirable. Stop- or off-times should be short, as 2 to 20 seconds, desirably 4 to 15 seconds.

The current densities should be maintained between 1 and 3 amperes per square inch, although values less than 1 and as high as 6 amperes per square inch can be used. At low current densities the powder is more dendritic, coarser and of a higher state of purity than at high current densities, all other factors being held constant.

The pH of the electrolyte can be as low as 2.0 and as high as 6.0. In general, other conditions being constant, the powders prepared at low pH's are coarser and tend to be dendritic while powders prepared at high acid pH's are finer and tend to be less dendritic or actually of a discrete, nodular or cuboidal nature.

The salts from which the electrolyte, for the production of the metal powders, is prepared include inorganic and/or organic metal salts. The anionic portion of the salt may be halides, preferably chloride, or sulphate, nitrate, phosphate, organic carboxylic acids such as aliphatic mono and polycarboxylic acids, sulfonic acids and mixtures thereof. Metal salts of these classes which are suitable electrolytes for the preparation of iron powder include for example the iron aliphatic and aromatic carboxylates, naphthenates, sulfonates, phosphates, sulfates, nitrates, chlorides, or mixtures thereof. Included among the metal carboxylates and sulfonates are the mono and poly aliphatic or aromatic carboxylates such as formate, acetate, propionate, lactate, glutarate, tartarate, citrate benzoate and salicylate; while the sulfonates include the aliphatic and aromatic sulfonates such as benzene sulfonate, hydroxybenzene sulfonate, toluene sulfonate, naphthalene sulfonate, alkylbenzene sulfonate, alkyl naphthalene sulfonate and mixtures thereof. Specific examples or preferred electrolytes are ferrous chloride, sulfate, acetate, formate, benzene sulfonate and mixtures thereof.

Powders have been prepared from solution having a concentration as low as 2 grams of ferrous ion per liter of solvent and as high as 25 grams of ferrous ion per liter of solvent. Saturated solutions may be used to prepare metal powder provided that the current density and pH have suitable values. Other conditions being constant the powders prepared at low concentrations are finer and of a more discrete nature than those prepared at higher concentration.

The electrolyte may be any suitable liquid medium. Suitable liquids include water, which is particularly preferred, alcohols, e.g. methyl, ethyl, or propyl alcohols, alkylene glycol, e.g. ethylene glycol, glycerol, ketones, pyridine, mixtures of water and alcohol and any similar conducting solvents.

The electrolyte of known and controlled composition, pH, and temperature may be pumped through the electrolytic cell in order to displace the spent electrolyte and to remove the powder from the cell and transport it to the collector. The spent electrolyte, after removal of the powder may be regenerated and re-used. The rate of flow of electrolyte must be commensurate with the sizes of the electrolytic cell and the total electrical current being passed through the cell.

The temperature of the electrolyte is generally maintained at about 25° C. However, temperatures as low as approximately the freezing point of the electrolyte and as high as 50° C. may be used.

The electrolytic cell may be of conventional construction. Since the current is reverse, the electrodes should desirably be of about equal area and desirably of the same and inert material, such as graphite, platinum, gold

and the like. Other materials such as copper, copper-amalgam, iron and the like may be used to produce powders. If desired, an electrode assembly of two or more electrodes of different metals may be used to produce combinations of metal powders such as nickel and chromium or iron and copper powder mixtures.

The metal powder after removal in a suitable manner from the spent electrolyte may be washed with any of various solvents or combinations. Most frequently the powder is first washed with deionized or distilled water, followed by an alcohol and/or acetone wash to remove water and finally washed with benzene in which the powder may be stored without appreciable oxidation especially if it is kept under an inert atmosphere.

The following examples are illustrative, parts being by weight unless otherwise indicated.

EXAMPLE II

An electrolytic solution consisting of water containing $\text{Fe}(\text{NO}_3)_3$ and HNO_3 and engine-block shavings (2.34 g. iron/l.) and having a pH of between 4.0 and 4.6 was pumped through a cell having symmetric graphite electrodes spaced $\frac{3}{16}$ " apart and 6" tall and approximately $1\frac{1}{2}$ " wide (electrode area being equivalent to 7 in.²) at a flow rate of 500 cc./min. A voltage of about 18.5 v. was applied in order to obtain a current density of 1.43 amperes/in.². The current was stopped every 30 seconds and reversed after a lapse of about 5 seconds. The pH of the spent electrolyte leaving the cell was 2.6. The spent electrolyte was regenerated and recirculated through the cell. The regeneration was accomplished by passing the electrolyte through a column containing engine-block shavings. The powder produced was recovered from the spent electrolyte in a magnetic trap. The powder was washed in water and then in acetone and finally washed and stored in benzene.

Electron microscope examination of the powder showed it to be nodular in shape and ranged in size from 0.05 to 0.25 micron in diameter and the average diameter was approximately 0.1 micron.

EXAMPLE III

Same procedure as given in Example 11, except that an aqueous electrolyte consisting of 50% nickel benzene sulfonate and 50% ammonium benzene sulfonate was pumped through a cell having symmetric graphite electrodes spaced $\frac{3}{16}$ " apart and having a total area of 9.5 in.² at a flow rate of 150 cc./min. The electrolyte contained 5.0 grams of nickel ion/liter. The pH was maintained at 4.2 to 4.4 and temperature at about 20° C. Current density was 1 ampere/in.² at 8 volts.

Electron microscopic studies show fine particles of about 0.1 micron and less in diameter. There was a tendency for the particles to agglomerate. Such submicron nickel powder was found to sinter at about 1100° F., and therefore produces a plate with a higher surface area, which is very useful for nickel cadmium batteries, or for catalytic applications.

When metal salts such as iron, chromium, copper or nickel or mixtures of iron and copper or mixtures of nickel and chromium toluene sulfonate, naphthalene sulfonate, nitrate, phosphate, or citrate, are used in the process of this invention, in which the pH of the electrolyte is maintained between 2 and 6, the current density kept between 1 and 5 amperes per inch square and the current reversed about every 30, 60 or 120 seconds, with stop- or off-time between current reversals being of from 2 to 20 seconds, powders and mixtures of powders and powder alloys of controlled shape and particle sizes of less than 1 and generally between 0.5 and 1 micron may be produced.

A number of further examples are given below, to illustrate various utilities of the invention in which submicron metal particles generally up to 1.0 micron in size and not usually over 3 microns are utilized either alone or in combination with metal particles of substantially larger

particle size, say not less than about 100 to 200 microns in particle size in which compositions unique properties are developed by the submicron metal particles of rhombic dodecahedral structure. Any pyrophoric properties in the submicron metal particles are eliminated or substantially removed before the pyrophoric material is admixed with other, non-pyrophoric material of the same or different substances or after such mixtures are made. Or other combinations of components or mixtures for special purposes of use, may inherently reduce or eliminate any pyrophoric properties. Various operations and articles are set forth below.

Thus compositions may be produced for use in the manufacture of electrical brushes of unusual conductivity and strength, or of powdered metal bearings and dies where segregation is undesirable, using for example the methods of powder metallurgy the composition containing metal powder of rhombic dodecahedral microparticle size of about 0.01 to 3.0 micron size selected from the group consisting of copper, tin, zinc, and non-metallic particles of carbon and graphite, and a lubricating oil non-reactive with any of the components present.

In the manufacture of carbon brushes, the so-called oil-less bearings, and similar molded products, metallic powders are blended and compressed, with or without heat, to obtain the desired molded objects. Various powders may be used, copper, tin, zinc, being among the most common.

In the manufacture of electrical brushes, where conductivity and strength are of prime importance, the presence of segregated zones of coarse and fine material is particularly objectionable. This is particularly true since most brushes are compounded of various ingredients of varying electrical conductivity, and electrical conductivity varies in different sections of the brush, and the tensile strength is of course that of the weakest zone.

In the manufacture of powdered metal objects such as bearings, the objections to segregation are no less striking. These objects are generally made by compressing the blended powder in dies, and then sintering. Segregation not only gives differences in physical properties due to zoning, but even more serious difficulties due to the formation of different alloys at different zones in the sintering operation.

The tendency of the metallic powder to separate can be substantially overcome, and the apparent density regulated, without destroying the flow of incorporating with the powder a small amount of a liquid which has a viscosity no higher than that of S.A.E. #10 lubricating oil (about 90 seconds Saybolt at 130° F.). For overcoming the tendency to separate it has been found that percentages of liquids between 0.0025 and 0.03 are effective in preventing segregation without stopping the flow. This same range also gives an effective control of the apparent density of the metal powder, although if apparent density control is the only consideration, smaller amounts will result in a reduction in apparent density over the untreated powder. Likewise if in the apparent density control flow is not important, further reduction in apparent density may be obtained if desired, by addition of the liquid up to 0.05% and even up to 0.1%. Ordinarily, however, no more than 0.05% will be added and the preferred range for both segregation and apparent density control is between 0.0025 and 0.03% of liquid based on the powder by weight.

Furthermore, we prefer to work with very thin liquids with viscosities of the order of kerosene, because as the viscosity increases, the ability to disperse throughout the mass lessens.

Another qualification which the liquid should possess is that it shall not react with the powder. For this reason, water, acids and unstable chlorinated hydrocarbons should not be used. While volatile liquids such as benzol, petroleum, ether, etc., function efficiently so long as they remain in the mass, a slowly evaporating liquid such as kerosene, however, remains in the powder during stor-

age, and evaporates from the molded article after manufacture; such a liquid is clearly preferable to a volatile liquid.

We have obtained elimination of segregation without loss of flow with volatile liquids like gasoline, benzol, petroleum ether, ethyl alcohol and ethyl acetate, with fixed liquids such as linseed oil, cottonseed oil and pine oil, and with slow evaporating liquids such as kerosene, petroleum naphtha and xylene.

As an example of our invention, we blended 90 parts of a copper powder of a mean particle size of 1-3 microns diameter with 10 parts of a tin powder of 1-3 micron size.

The copper-tin mixture was then treated with kerosene. As little as .0025% addition of kerosene practically eliminated the tendency toward segregation and that above 0.03% there was a total loss of flow, and that for best results in both flow properties and prevention of segregation the amount should not be over 0.01%.

A copper powder-carbon mixture, similarly treated with 0.01% kerosene, gave similar improved results over an untreated mixture.

A magnetic tape carrying a magnetic record recorded thereon may be produced in conventional manner from iron particles of rhombic dodecahedral structure of particle size between about 0.01 and 3 microns treated to make them non-pyrophoric. Methods conventionally employed to produce such tapes include the following. The tape is conventionally moved under a box containing iron particles of character referred to immediately above, so that the iron particles align themselves in accordance with the position and degree of magnetism in the tape. These iron particles of submicron size are highly sensitive to, and readily controlled to insure few, if any, substantial defects. An antifriction bearing may be made provided with a bearing surface consisting essentially of a thermally set synthetic resin having dispersed therein a transition element bearing metal of rhombic dodecahedral microparticle from about 0.1 to 3 microns in size and in amount of from about 5 to 50% by weight of the composition of the bearing. The bearing metal may, for example, be copper, tin or zinc, of particle size and internal structure as set forth above. Conventional components like flakes graphite or flaked lead may be included. The resin may be a phenol formaldehyde type, melamine resins, etc. available on the market. The following is an exemplary composition:

	Grams
Micron copper powder non-pyrophoric, as above	
1-3 microns	3.5
Flaked graphite	5.0
Phenolformaldehyde resin powder	5.0

The above ingredients are homogeneously mixed together and may be molded under pressure and heat according to known plastic molding procedure, to form masses of the desired shape. Heat treatment is applied during or after molding to thermally set the resin, thus providing a body of considerable mechanical strength, which can be trimmed or shaped by known finishing methods, such as grinding.

A flushing oil may be produced comprising a petroleum oil solvent for hydrocarbon lubricating oils and their decomposition products in which is incorporated from a minor amount of for example, about 0.1 to about 5% to larger amounts such as 5 to 10%, by weight of an engine conditioning material of a transition metal powder of rhombic dodecahedral sub-microparticle such as 0.1 to 1.0 micron or even up to 3 microns in size, the metal components being non-pyrophoric, particularly when selected from copper, tin, zinc and mixtures thereof. An example of a composition is submicron copper 0.2%, flushing oil viscosity 30 to 210° F. Saybolt 99.8%. Another example is submicron tin 1.0%, Edelenan extract (25-35 viscosity Saybolt at 210° F.) 99%.

Having thus set forth my invention, I claim:

1. An antifriction bearing having a bearing surface consisting essentially of a thermally set synthetic resin of phenol formaldehyde or melamine resins and dispersed therein a transition element bearing metal of rhombic dodecahedral microparticle from about 0.01 to 3 microns in particle size and in amount of from about 5 to 50% by weight of the composition, the composition being inherently non-pyrophoric, the metal having been pyrophoric when produced.

2. A composition for production of antifriction bearings consisting essentially of a thermally set synthetic resin of phenol formaldehyde or melamine resins and dispersed therein a transition element bearing metal of rhombic dodecahedral microparticle from about 0.01 to 3 microns in particle size and in amount of from about 5 to 50% by weight of the composition, the composition being inherently nonpyrophoric, the metal having been pyrophoric when produced.

3. A composition as set forth in claim 2 in which the metal is selected from the group consisting essentially of copper, zinc, tin, and mixtures thereof.

4. An antifriction bearing as set forth in claim 1 in which the metal is selected from the group consisting essentially of copper, zinc, tin, and mixtures thereof.

5. A flushing oil comprising a petroleum oil solvent for hydrocarbon lubricating oils and their decomposition

products in which is incorporated from a minor amount of about 2 to 10%, by weight of an engine conditioning material of a transition metal powder of rhombic dodecahedral microparticle structure not exceeding about 3 microns in size, the metal powder being inherently non-pyrophoric, the metal having been pyrophoric when produced.

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