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3,583,864

CHEMICAL PROCESS OF PRODUCING AN IRON-COPPER ALLOY POWDER

Filed May 5, 1969

2 Sheets-Sheet 1

FIG. 1

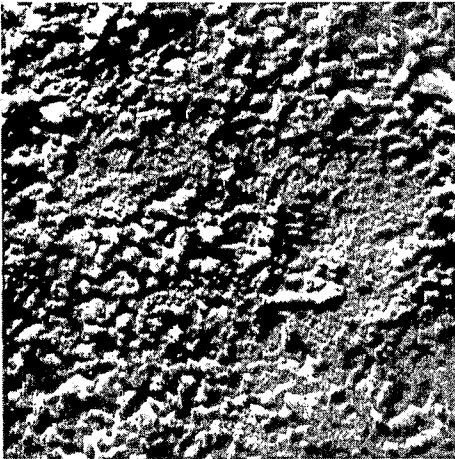
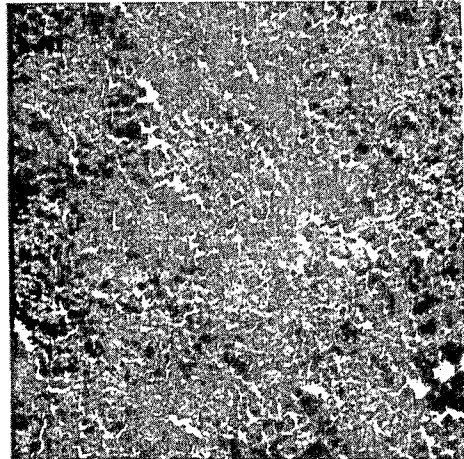


FIG. 2



0.1 MICRON

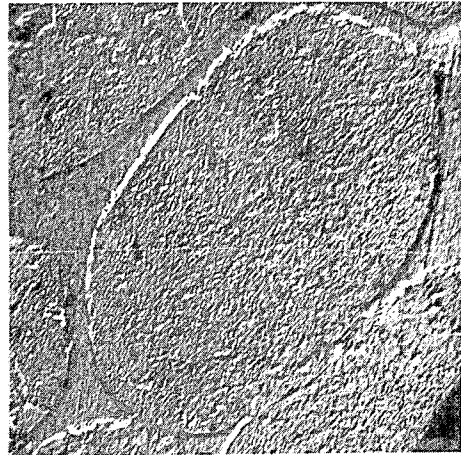
FIG. 3



10.0 MICRONS

0.1 MICRON

FIG. 4



10.0 MICRONS

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FIG. 5



1.0 MICRON

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CHEMICAL PROCESS OF PRODUCING AN IRON-COPPER ALLOY POWDER

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Continuation-in-part of application Ser. No. 471,767, July 13, 1965, now Patent No. 3,489,548. This application May 5, 1969, Ser. No. 821,935

Int. Cl. B22f 1/00, 9/00

U.S. Cl. 75—5BA

21 Claims

ABSTRACT OF THE DISCLOSURE

A particulate alloy of iron intimately infiltrated with from about 1 to about 50% by weight of copper is produced by mixing a reducible compound of iron with an appropriate proportion of copper compound selected from the group consisting of elemental copper and reducible compounds of copper, the mixture being heated under reducing conditions at a temperature between about 1010 and 1150° C. until the reducible compounds are substantially completely reduced to the metal state.

CROSS REFERENCE TO RELATED APPLICATIONS

This application is a continuation-in-part of application Ser. No. 471,767 filed July 13, 1965, now Patent No. 3,489,548.

BACKGROUND OF THE INVENTION

This invention is concerned with alloys of copper and iron, and more particularly with novel iron powders pre-infiltrated with copper, and with processes for their preparation.

As employed herein and in the appended claims, an alloy is defined to be a substance having metallic properties and composed of two or more chemical elements of which at least one is elemental metal. It has in the past been recognized that the physical properties of iron are improved by alloying with copper. Since these metals are virtually insoluble in each other at room temperature, and since their mutual solubility is quite limited even at elevated temperatures, various expedients have been employed in an attempt to bring them into intimate association. Thus, copper and iron powders have been blended and molded into finished objects by the techniques of powder metallurgy. In addition, the infiltration process has been employed, i.e. filling the pores of a sintered powder metallurgy part, of iron or steel in this case, with a metal or alloy of lower melting point, e.g. copper.

These prior methods for the preparation of copper-iron alloys are characterized by important problems and disadvantages. Blended copper and iron powders are subject to segregation in storage and shipment. More important, even very finely divided, blended powders do not provide the degree of homogeneity which affords optimum properties. In addition, sintering of compacted copper-iron powder blends results in expansion of the compact, representing a porosity increase which is a major factor in inferior strength, molding flaws and high rejection rate.

A relatively simple procedure has now been discovered for the preparation of novel copper-iron alloys in powder form, offering substantial cost advantages over conventional infiltration techniques. This procedure imparts to the new powders a unique microstructure, characterized by a more intimate admixture of the elements, to which the superior physical properties which result may be attributable. Upon simple pressing and sintering, the new

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particulate alloys shrink to high-density products of great strength.

SUMMARY OF THE INVENTION

In essence, the process of the present invention entails blending a reducible iron compound with copper or with a reducible copper compound, and reducing these reactants to the metallic state at elevated temperature. The relative proportions are selected to yield a product containing from about 1 to about 50% by weight of copper.

DESCRIPTION OF THE FIGURES

The distinctive metallographic features of the new alloys will be better understood by reference to the accompanying illustrations, which are photographic reproductions of highly magnified electron microscope photomicrographs. Each photograph depicts a portion of a single particle of one of the new alloys, as etched in a 3% solution of nitric acid in alcohol in order to erode the copper-rich areas.

FIG. 1 illustrates an alloy of iron with 7% by weight of copper, prepared by the process of the present invention and enlarged approximately 100,000 diameters.

FIG. 2 shows the alloy of FIG. 1 after heat treatment as detailed hereinafter, also enlarged approximately 100,000 diameters

FIG. 3 depicts an alloy of iron with 20% by weight of copper, prepared in accordance with the present invention and enlarged approximately 3600 diameters.

FIG. 4 shows the alloy of FIG. 3 after heat treatment, also enlarged approximately 3600 diameters.

FIG. 5 shows a portion of the particle depicted in FIG. 4, this time enlarged approximately 17,700 diameters.

The salient features of these illustrations will be referred to hereinafter, in connection with a discussion of the microstructure of the new products.

DETAILED DESCRIPTION OF THE INVENTION

Copper sinters at about 825–850° C., and it melts at about 1083° C. in the pure state and at about 1095° C. when it contains 3.2% or more of iron. In order to achieve the uniquely intimate consolidation of the iron and copper which characterizes the invention, it is essential that the temperature during the reduction step reach at least 100° C. above the sintering temperature of copper. In addition, it has been found that inferior results are achieved at reduction temperatures below about 1850° F. (1010° C.). Such reduction conditions produce a powder alloy of low apparent density containing excessive fines and characterized by poor flow and frequently by high shrinkage upon pressing and sintering. Further, and particularly in reduction with carbon (e.g. coke), temperatures below 1850° F. require excessive time for adequate reduction or lead to substantially incomplete reduction. The minimum practical and economic reduction temperature is 1850° F.

However, particularly for those alloys containing in excess of 20% by weight of copper, reduction temperatures at or above the copper melting temperature tend to cause agglomeration of the reduced product to a mass which can be broken down into smaller particles only with difficulty. Accordingly, especially when alloys of such high copper content are prepared, it is best to keep the reduction temperature below the melting point of copper. In all cases, however, reduction temperatures above about 1150° C. are undesirable, since they favor extensive product agglomeration, producing a tough, hard mass which is impossible to grind to metal powder by any economic means. Best results have been achieved at 1850–2000° F. (1010–1093° C.) and especially at 1900–1950° F. (1038–1066° C.).

Under the described conditions, the reactant mixture is reduced to a particulate alloy of iron intimately infiltrated with copper, without the need for the copper to flow through the mass of iron to saturate the pores as is the case in conventional infiltration of molded iron compacts. During the new process, the oxide or other reducible iron compound undergoes a solid state reduction. Where the reduction is conducted below 1094° C., but at least 100° above the copper sintering temperature, the copper does not liquify, but the reduced solids undergo grain boundary diffusion, with comparable results. These lower reduction temperatures are especially preferred in the production of alloys of high copper content, as previously discussed, and the high copper volume fractions are believed to favor products of infiltrated nature.

When reference is made in this disclosure and in the appended claims to particulate alloys of iron infiltrated with copper, this expression is intended to describe individual particles containing both copper and iron in intimate mutual dispersion, as distinguished from mere blends of copper powder with iron powder, and as further distinguished from particles of the one element merely coated with the other.

By their nature, the new particulate alloys of the present invention are not subject to segregation into the individual elements in storage or shipment. They may be truly termed "preinfiltrated" alloys, since they are directly moldable by conventional powder metallurgy techniques to useful parts, without the need for a separate penetration of liquid copper into a molded iron part as practiced in conventional infiltration.

Any reducible iron compound may be employed in the new process, including iron salts or any oxide of iron such as hematite, magnetite, beneficiated magnetite ores, flue dusts, synthetic oxides or reducible mill scales, e.g. from rolling mill operations. By reducible mill scales is meant those which are reducible to the extent of about 99% or better, such as carbon steel mill scale and low alloy steel mill scales.

The source of copper may be elemental copper, such as reduced copper powder, atomized copper, electrolytic copper powder or hydrometallurgical copper powder. However, it will usually be less expensive to employ an oxide of copper, either precipitated or mechanically produced, such as cuprous oxide, cupric oxide, copper mill scale, copper flue dust, or cement copper, a by-product of mine waste water which typically contains about 50-98% cuprous oxide. Where copper oxides are employed, it is sometimes beneficial to incorporate nitric acid in the reaction charge, to promote a more intimate dispersion. Alternatively, cupric nitrate or other water-soluble copper salt, may be employed in water solution as the copper source.

In addition to copper and iron, nickel, cobalt, molybdenum or tungsten may be included, or reducible compounds of these elements, such as nickelous acetate, cobaltic oxide, molybdic oxide, or tungstic anhydride. Such elements will generally be employed in minor proportion, i.e. up to about 6% by weight of any one element or a total of up to about 12% by weight in the case of several in combination, in order to improve strength. Such elements may be added in the form of soluble salts such as nitrates or acetates, for optimum dispersion, or as compounds in combination with an acid or base solvent, such as ammonia water or nitric acid.

Those reactants are preferred which have particle sizes finer than 250 microns, and especially preferred are iron compounds finer than about 50 microns and copper compounds finer than about 20 microns, since these favor the most intimate interdispersion of the elements.

While not essential, it is often advantageous to blend the reactants with an aqueous dispersion of an organic adhesive, to minimize dusting losses or segregation of the reactants during handling and subsequent processing.

Suitable binders include animal protein glue, methyl cellulose, hydroxyethyl cellulose, carboxymethyl cellulose, sugar and the like. Such compounds are readily decomposed at the reduction temperature and should preferably be low in ash content. Adhesive levels of about 0.5% of the total batch weight are usually entirely adequate, the proportion of water usually being up to about 18% of the total batch.

Where an adhesive is employed, the blending of reactants may be advantageously conducted in a mix miller or "chaser," which will form the ingredients into hard pellets up to about an inch in diameter with little or no secondary grinding. These pellets may be charged to the reduction furnace without drying. If no adhesive is employed, the reactants are thoroughly blended before charging to the furnace.

Reducing conditions may be provided within the furnace in the form of a gaseous reducing atmosphere, e.g. hydrogen or carbon monoxide, or sources of such agents including dissociated ammonia, water gas, producer gas and the like. Alternatively, the reducing conditions may be provided by incorporating finely divided carbon within the reactant mixture, and this procedure is preferred for economic reasons. Suitable forms of carbon include lamp black, petroleum coke, anthracite fines, carbon black, bone black or graphite. The minimum carbon levels, being dependent on the particular reactants and reactant proportions, may be estimated from the stoichiometry of the reaction, but optimum levels are best determined by experiment. Carbon levels of about 8-12% are typical. Even where a gaseous reducing atmosphere is provided, it is sometimes beneficial to incorporate a minor proportion of carbon, particularly in large batches, to ensure effective reduction. As is well known in the reduction art, where carbon is the principal reducing agent it is ordinarily desirable to sweep the furnace with a carrier gas, to drive off the volatile reduction products and prevent re-oxidation. Suitable carrier gases include endothermic gases high in nitrogen, exothermic gases, methane, propane, natural gas, or the like. Manufactured gas, producer gas or hydrogen may, of course, also be used if desired.

The reduction is continued until the reducible compounds are substantially completely reduced, i.e. about 93-98% reduced. During reduction, the product particles usually form a lightly sintered sponge, which may readily be subdivided to final particle size specifications by milling. It has been found that a residual oxygen content of 2% or more yields a particularly friable sponge which is broken up more easily than sponges of lower oxygen content. The time required to carry the reduction to this level will vary with the reducing conditions, the batch size, and the particular reactants selected. Ordinarily, reduction is substantially complete on a laboratory-size batch in from about 30 minutes to two hours. It is undesirable to prolong the reduction unnecessarily, e.g. to the point of quantitative oxygen removal, since this may lead to difficulty in breaking up the sponge.

After cooling, the product may be ground in a hammer mill or pulverizer to final particle size specifications, generally to pass 60 or 80 mesh.

The resulting powders may be molded into useful shapes by conventional powder metallurgy techniques, i.e. by compacting at about 10-60 tons per square inch and then sintering under non-oxidizing conditions at elevated temperature, preferably at or above the melting point of copper for optimum properties. It should be noted that where blended copper and iron powders are molded in this fashion, it is necessary at high copper content to sinter below the melting point of copper, with attendant strength disadvantages, in order to avoid loss of copper by "blistering." This is caused by agglomeration of the copper-rich masses in such blends during sintering. It is also important to note that the sintering of premixed copper and iron powders results in an expansion of the

compact as a reflection of the alloying process. In the new particulate alloys, on the other hand, any expansion as a result of alloying occurs during the reduction step and not during the sintering of the compacted particles. The sintering of these new alloys represents a consolidation of the pre-infiltrated or alloyed particles. This results in shrinkage of the sintered compact, which, as previously noted, is more desirable.

Sintered parts prepared from the particulate alloys of the described reduction process possess good physical properties even without further treatment. The degree of shrinkage and the resulting strength depend mainly on the copper content, with maximum shrinkage and maximum strength at about 30% copper.

A further important feature of the present invention resides in the discovery of a novel heat-treatment process for the alloy powders, which leads to a striking and unprecedented increase in the density and physical strength of the final sintered compact. This heat treatment represents in effect a densification or "preshrinkage," reducing the degree of shrinkage in the final sintering step without, however, leading to the undesirable expansion effect referred to previously.

The α - γ transition temperature of iron lies at about 910° C., but in the case of iron containing over 3.5% copper this transition occurs at about 835-850° C. It has been found that heat treatment of the new alloy powders is most effective at a temperature between the α - γ transition temperature of the iron-rich phase and a temperature about 150° C. lower. The best results are obtained by heat treating at a temperature less than 50° C. below the α - γ transition, preferably at about 825-845° C. Such conditions lead to development of the highest density, strength and hardness, with minimum shrinkage, upon sintering. Properties begin to drop off abruptly from the optimum when the heat-treatment temperature exceeds 845-850° C.

For best results, the powder should be heat treated for at least about 5 minutes before cooling. This should afford sufficient time for the major portion of the excess copper within the supersaturated iron to separate as a fine precipitate. The maximum time is not critical, and periods up to 10 hours may be used. However, there is no advantage to heat treating for more than 4 hours. Of course, some time may be needed to heat the batch to the operating temperature, and this will vary with the size of the batch.

If the reduced powder subjected to heat treatment contains over 2% oxygen, it is best to heat treat under reducing conditions, so as to further reduce oxygen content below 2% and preferably below 1% during this step. If, however, the oxygen content is already 2% or less, an inert atmosphere may be employed if desired.

The powders are discharged from the furnace as loosely sintered masses, which may be re-ground to particles finer than 60-80 mesh in the same manner as after the primary reduction process.

Heat treatment as described greatly enhances the properties of the alloy powders of this invention. After compacting and sintering, the products exhibit outstanding tensile strength, as may be seen from the appended examples. It should be noted that those alloys containing about 1-6% copper have tensile strengths above 45,000 p.s.i.; while 6-10% copper yields 60,000 p.s.i. or more; 10-40% copper yields 75,000 p.s.i. or more; and 40-50% copper yields, tensile strength above 65,000 p.s.i. By way of comparison, sintered parts made from preblended powders have tensile strengths which level off at 40-45,000 p.s.i., and this is substantially unaffected by prior heat treatment of the powder blends.

In addition, sintering shrinkage is greatly reduced as a result of heat treatment of the new alloys, density is substantially increased, and porosity reduced. Each of these advantages is illustrated quantitatively in the examples.

Such sintered properties are within the range of or superior to those of the convention infiltrated part, yet they are obtained by the rather simple and cheap pressing and sintering process rather than by the more involved and costly infiltrating technique.

A preliminary insight into the microstructures of the new alloys is provided by their X-ray diffraction patterns. An alloy containing 20% copper displays essentially the same pattern after primary reduction and after heat treatment: both an α -iron-rich phase and an ϵ -copper-rich phase is detected. The diffraction pattern of a 7% copper alloy after heat treatment is substantially similar, except for the expected indication that a lower proportion of the copper-rich phase is present. However, the 7% alloy after primary reduction exhibits a pattern corresponding to a single α -iron-rich phase. In each of these diagrams, the iron peaks are relatively flattened, the copper peaks sharper.

The failure to resolve a second phase by X-ray diffraction in the 7% copper alloy after reduction is rather surprising, indicating that alloys of copper content below the solubility limit in iron at the reduction temperature (about 8% at 1125° C.) retain the copper in solution after primary reduction. This may be related to the rate of cooling upon removal from the furnace, the temperature dropping to about 490° C. in the first minute in the typical case, thereby quickly traversing the range of maximum solubility change. (The solubility of copper in iron at 500° C. is only about 0.25%.) On the other hand, X-ray data show that subsequent heat treatment within the specified temperature range for 5 minutes or more does result in precipitation of an X-ray resolvable copper-rich phase.

A more accurate insight into the microstructures is gained by microscopic examination of the alloy particles, mounted, polished, and etched with nitric acid to erode copper-rich areas and thereby delineate the phases.

In those alloys containing from about 1 to 10% copper the microstructure is seen to be highly uniform, with the copper content up to about 8% dispersed within the iron grains in the form of particles less than about 1000 Å. (0.1 μ) in diameter. At 250 power magnification, no clear-cut grain boundaries can be seen, and it is necessary to turn to the higher magnification of the electron microscope for further delineation. In FIG. 1 is shown a 7% copper alloy of iron after primary reduction, and FIG. 2 shows the same alloy after heat treatment, both views enlarged about 100,000 diameters. The depressions represent areas where copper was removed by the etching acid, and the fineness of the dispersion is readily apparent. When the fields are surveyed further, it is found that the specimen after reduction displays dispersed copper-rich particles about 350 Å. in diameter arranged in clustered bands, with an average distance of about 150 Å. between copper particles. The clusters are separated from each other by distances ranging from as little as 500 Å. to as great as about 0.75 micron. Between the clusters, copper-rich particles also averaging about 350 Å. in diameter are detected, but it is likely that particles smaller than the 50 Å. resolution limit are also present. It is likewise possible to detect cracks in the microstructure, about 0.1-0.3 μ in length by 400-900 Å. in width, as well as angular voids 0.1-0.9 μ in diameter. Upon heat treatment, the cracks are no longer visible, and the clusters now appear in bands or rings about 10 μ ±5 in length by 3 μ ±1 in width. The copper particles in these rings are found to average about 400 Å. in diameter, with an interparticle spacing of about 150 Å. Those copper particles between clusters which are resolved average about 175 Å. in diameter, with an interparticle spacing of about 550 Å.

Microscopic examination of those alloys of the present invention which contain in excess of 10 and up to about 50% by weight of copper reveals that they contain unusually fine and closely packed iron-rich grains, less than about 35 μ in their major or largest diameter, with an average separation between adjacent iron grain boundaries of

less than about 5 μ , in a primary copper-rich matrix. These iron-rich grains contain a highly uniform dispersion of finely divided copper-rich particles.

FIG. 3 illustrates an alloy containing 20% copper, after primary reduction, and FIG. 4 depicts the same alloy after heat treatment, each enlarged about 3600 diameters. In these pictures, the prominent insular areas which occupy most of the field of view are plateau-like iron grains surrounded by copper-rich "valleys" eroded by the acid. There may be a tendency here for the iron grains to appear to the eye as depressions, but this apparent stereoscopic reversal is an optical illusion which can be attributed to the lighting. It will be noted that in the specimen after primary reduction, the iron grains are irregular in shape, whereas after heat treatment they have become rounded or spheroidized.

At high magnification, cracks are detected in the reduced specimen, about 2500 A. in length and 850 A. in width. These may account at least in part for the higher shrinkage which occurs upon sintering, and they are absent in the particles after heat treatment.

The following are the average dimensions observed in the 20% copper alloy after primary reduction at 1125° C., as determined by Zeiss counter measurements:

Iron grains:	μ
Length -----	17.50
Width -----	13.89
Intergranular spacing -----	1.76
Copper particles within iron grains:	
Length -----	0.0423
Width -----	0.0371
Interparticle spacing -----	0.0352
Iron particles in copper-rich areas:	
Length -----	0.0268
Width -----	0.0216
Interparticle spacing -----	0.0894

Upon close inspection, the iron grains of FIG. 4 present a much rougher texture than those of FIG. 3, the reason for which is revealed by greater magnification. FIG. 5 represents a portion of the field of view of FIG. 4, enlarged about 17,700 diameters. The most prominent feature of the illustration is an elongated "valley" or copper-rich area separating portions of two adjacent iron grains. The pictured area within those iron grains is pitted by erosion of copper-rich particles through the acid treatment, and it is seen that the copper particles within the iron grains fall into two different size groups: primary particles having a diameter of about 0.1-0.5 μ , and secondary particles having a diameter less than about 0.05 μ .

This unexpected and unique structure only appears after heat treatment, and it seems likely that the larger primary particles represent agglomeration of the original copper particles in the reduced specimens, resulting from the difference in solubility of copper in iron at the reduction temperature (about 8% at 1125° C.) and at the heat-treating temperature (about 1.4% at 835° C.). It may be theorized that the primary copper particles represent the copper in excess of solubility at the heat treating temperature, which agglomerates during such treatment step. It may further be theorized that the smaller secondary particles represent that copper which dissolved in the iron to saturation at the heat-treating temperature and subsequently separated during cooling to room temperature. The fact that these secondary particles are similar in size to the copper particles present within the iron grains after primary reduction lends credence to this hypothesis. Nevertheless, it should be understood that the present invention is not limited by any theory or hypothesis, however plausible.

The following are the average dimensions observed in the 20% copper alloy after heat treatment at 835° C.

Iron grains:	Microns
Length -----	20.09
Width -----	16.39
Intergranular spacing -----	1.66

Primary copper particles within iron grains:

	Microns
Length -----	0.2713
Width -----	0.2404
Intergranular spacing -----	1.66
Secondary copper particles within iron grains:	
Length -----	0.0331
Width -----	0.0254
Interparticle spacing -----	0.0287
Iron particles in copper-rich areas:	
Length -----	0.1238
Width -----	0.1122
Interparticle spacing -----	0.4720

The dispersion of more copper within the iron grains in the alloys of the present invention may account for their higher sintered strength relative to conventionally infiltrated alloys of equivalent density, whose copper content would appear to be more concentrated between the grains.

Microscopic examination of the novel alloys of this invention after compacting and sintering has also been conducted, and it is found that the alloys as sintered closely resemble the unsintered particles. This can be seen by comparing the average iron grain sizes and intergranular distances in the heat-treated powders and the sintered parts which result, e.g. for the 20% copper alloy;

	Iron grain		Distance between iron grains, μ
	Length, μ	Width, μ	
Heat-treated powder -----	20.09	16.39	1.66
Sintered part -----	22.09	17.39	1.68
Conventional infiltration -----	56.66	27.91	8.33

The resistance to increased grain growth during sintering, as evidenced above, would seem to reflect the structural stability of the new alloys. The finer, more closely packed grain structure in contrast to the product of conventional infiltration is also notable in the above table. This contrast is further illustrated by a comparison of alloys containing 40% copper:

	Iron grain		Distance between iron grains, μ
	Length, μ	Width, μ	
Sintered part from heat treated powder -----	16.28	14.00	3.41
Conventional infiltration -----	40.01	22.66	12.51

The porosity of sintered iron compacts is ordinarily such that it is necessary to introduce in excess of 10-15% copper for adequate infiltration by conventional technique. Accordingly, for a standard of comparison for the new alloys of low copper content, it is necessary to turn to sintered parts made from blended copper and iron powders. Sintered bars containing 7% copper and prepared from a blend of 100 mesh copper and iron powders, when microscopically examined, exhibit large angular pores and massive copper areas 30 μ and more in diameter. The sintered compact prepared from the new particulate alloy containing 7% copper, on the other hand, exhibits a very fine, uniform, close-packed structure.

The excellent physical properties provided by the new particulate copper-iron alloys can be even further enhanced by various techniques, providing tensile strengths as high as 150,000 p.s.i. For instance, the incorporation of minor proportions of graphite before molding and sintering affords increases of from 30,000 to 60,000 p.s.i. in tensile strength. Graphite levels of about 0.5-2% are usually adequate. Re-pressing and re-sintering (coining) operations are also beneficial for increasing density and

strength, as are various post treatments, such as quenching, drawing and normalizing, as further illustrated in the examples which follow.

Provision of the following examples for illustrative purposes is not intended to restrict the invention, the scope of which is defined by the appended claims.

Example 1.—7% copper alloy

(A) Reduction:	Grams
Iron mill scale	1247.1
Dried cement copper	77.0
Hydroxyethyl cellulose	5.3
Water	285.0

The iron mill scale of the above formulation is a by-product of steel blooming or finishing mills, finer than 325 mesh with about 50% $\pm$ 5% coarser than 20 microns. It has an apparent density of 1.8–2.2 grams per cubic centimeter and an analysis as follows:

	Percent
Fe	¹ 74.58
O	24.58
Mo	<0.05
Mn	0.308
Ni	0.038
C	0.006
S	0.027
P	0.100
SiO ₂	0.301
Al ₂ O ₃	0.010

¹ As FeO 70.50%.

The cement copper of the above formulation is a by-product of mine waste water, finer than 20 microns with about 85% finer than 10 microns. It has an apparent density of 0.8–1.5 grams per cubic centimeter and an analysis as follows:

	Percent
Cu	¹ 90.80
O	8.03
Zn	0.22
Fe	0.47
SiO ₂	0.03
Other metals	0.26
Soluble nitrates	0.01
Soluble chlorides	0.08
Soluble sulfates	0.10

¹ As Cu, 2.91; as Cu₂O, 96.72; as CuO, 2.61.

The ingredients are combined and milled into pellets in a mix muller or chaser, which permits intimate admixture with a minimum of grinding action. The resulting pellets are charged to a reduction furnace at about 1120–1135° C. and held at that temperature in hydrogen or dissociated ammonia for 45 minutes. After reduction the pellets are removed from the furnace and broken up, first in a hammer mill to $\frac{1}{8}$ inch and smaller, and then in a micropulverizer so that all particles are finer than 80 mesh. The product has an apparent density of 2.3–2.5 grams per cubic centimeter and an oxygen content of about 1.6% (obtained by reduction in hydrogen at 1050° C. for 30 minutes) or 2.37% (obtained by Leco method—melting in vacuum at 3500° F.). The hydrogen weight loss reflects only reducible oxygen content.

Hematite (Fe₂O₃) or magnetite (Fe₂O₃·FeO) in sufficient quantity to provide the same iron content may be substituted for the iron mill scale in the above formulation. In the same way, pure cuprous oxide may be substituted for the cement copper.

For the reduction step, carbon monoxide may be substituted for hydrogen, or gases rich in carbon monoxide or hydrogen, such as producer gas, may be used.

(B) Heat Treatment:

150 grams of the reduced powder is charged to the reduction furnace, maintained at 825–845° C. for one

hour, and cooled. The powder is discharged from the furnace as a loosely sintered mass and reground to powder finer than 80 mesh in the same manner as after the primary reduction. The annealed powder has an oxygen content of 0.3% (by weight loss in hydrogen) or 1.14% (Leco method).

Example 2.—1 and 2% copper alloys

	Grams
Iron mill scale	1314.2
Cupric nitrate trihydrate	37.5 or 76.0
Carboxymethyl cellulose	6.2
Water	300.0

The above formulations are reduced at 1150° C. and heat-treated in the same manner as is described in Example 1. Hematite (Fe₂O₃) or magnetite (Fe₂O₃·FeO) in sufficient quantity to provide the same iron content may be substituted for the iron mill scale.

The cupric nitrate may be replaced by equivalent proportions of cupric oxide and nitric acid, or by an equivalent proportion of cupric acetate.

Example 3.—14% copper alloy with 1% nickel

	Grams
Iron mill scale	1153.3
Dried cement copper	154.0
Animal protein glue	6.0
Water	270.0
Nickel nitrate (20.3% Ni)	49.5

This formulation is pelletized, reduced at 1110° C. and heat-treated as described in Example 1. Equivalent quantities of hematite (Fe₂O₃) and nickelous acetate tetrahydrate may be substituted for the iron mill scale and nickel nitrate. An equivalent proportion of pure cuprous oxide may be substituted for the cement copper.

Example 4.—20% copper alloy

	Grams
Iron mill scale	1072
Dried cement copper	205.2
Cupric nitrate trihydrate	37.1
Lampblack	105.1
Methyl cellulose	7.9
Water	153

These ingredients are pelletized, charged to a reduction furnace, and maintained at 1095° C. for 75 minutes while sweeping with a lean endothermic carrier gas (approx. 84% nitrogen, 10% carbon monoxide, 6% hydrogen). After reduction, the charge is processed by milling and heat-treating as detailed in Example 1.

A 20% copper alloy containing 1% molybdenum is prepared from the following ingredients by the reduction and heat treatment procedure of Example 1:

	Grams
Iron mill scale	1072.8
Dried cement copper	220.5
Molybdc oxide	15.0
Ammonia water (26 Bé.)	34.0
Carboxymethyl cellulose	6.0
Water	275

A similar alloy containing 1% tungsten is prepared in the same manner, by substituting 12.6 grams of tungstic anhydride (WO₃) for the molybdc oxide in the above formulation. An equivalent cobalt content is provided by substituting 14.1 grams of cobaltic oxide (Co₂O₃) for the molybdc oxide. Hematite (Fe₂O₃) may also be substituted for the iron mill scale by appropriate adjustment in the quantity added.

Example 5.—Particle size effect

The procedure of Example 1A is repeated with iron mill scales of varying particle size. The resulting reduced powders are compacted at 50 tons per square inch, sintered in

hydrogen at 1120° C. for 45 minutes, and subjected to physical testing, with results as follows:

Mill scale finer than.....	250μ	44μ	20μ
Mill scale, average particle size.....	80μ	20μ	5μ
Held products:			
Linear shrinkage, percent.....	2.70	1.81	2.23
Sintered density, g./cc.....	6.25	5.96	6.21
Tensile strength.....	30,200	39,600	24,000
Elongation, percent.....	1.4	1.0	1.6
Hardness.....	B 45.3	B 34.0	B 48.0

Example 6.—Binder effect

The procedure of Example 1A is repeated both with and without the inclusion of the hydroxyethyl cellulose adhesive. In a second pair of experiments, this procedure is repeated with adjustment of the cement copper level to provide 20% copper alloys prepared both with and without adhesive. Each of the four reduced powders is compacted at 50 tons per square inch, sintered in hydrogen at 1120° C. for 45 minutes, and subjected to physical testing, with results as follows:

	7% Cu		20% Cu	
	With binder	Dry mix	With binder	Dry mix
Tensile strength, p.s.i.....	39,600	33,200	60,500	51,000
Sintered density, g./cc.....	6.96	5.67	6.87	6.54
Elongation, percent.....	1.0	0.5	3.0	2.0
Linear shrinkage, percent.....	1.81	1.025	3.68	2.65
Hardness.....	B 32.3	B 28.0	B 69.6	B 51.2

¹ Expansion.

Example 7.—Reduction temperature effect

The procedure of Example 1A is repeated in a series of batches with the single exception that some of these are reduced at 1000° C. instead of 1120–1135° C. Each of the reduced powders is then compacted at 50 t.s.i., sintered in hydrogen at 1120° C. for 45 minutes, and subjected to physical testing, with results as follows:

	Reduction temperature					
	1,000° C.			1,125° C.		
	Batch			Batch		
	1	2	3	1	2	3
Reduced powder:						
Apparent density, g./cc.....	2.15	1.83	1.73	2.35	2.39	2.44
Flow rate, sec./50 g.....	35.4	40.0	None	30.7	29.3	29.9
—325 mesh, Percent.....	42.2	59.8	67.9	32.8	36.7	42.8
H ₂ wt. loss, Percent.....	1.70	1.93	2.07	1.21	1.22	1.29
Compact: green density, g./cc.....	6.40	6.29	6.33	5.76	5.67	5.73
Sintered compact:						
Sintered density, g./cc.....	6.31	6.20	6.09	5.96	6.02	6.04
Tensile strength, p.s.i.....	40,900	32,200	40,000	39,600	40,100	39,500
Elongation, Percent.....	1.3	1.4	1.0	1.0	1.1	1.3
Linear shrinkage, percent.....	2.00	2.013	2.014	1.81	1.75	1.82
Hardness.....	B 55.3	B 50.7	B 48.0	B 32.3	B 30.7	B 34.0

¹ 1,100° C. for 30 minutes.

² Expansion.

Example 8.—Heat-treatment effect

A quantity of the reduced 7% copper alloy powder prepared in Example 1A is divided into a number of 150 gram batches for heat treatment as described in Example 1B, but the temperatures and periods of heat treatment are varied as outlined below. Each heat-treated powder

is compacted and sintered as described in Example 7, and tested with results as follows:

	Heat-treated at 850° C.						Heat-treated at 700° C.						Heat-treated at 600° C.					
	1 hr.			2 hrs.			1 hr.			2 hrs.			1 hr.			2 hrs.		
	1 hr.	2 hrs.	3 hrs.	1 hr.	2 hrs.	3 hrs.	1 hr.	2 hrs.	3 hrs.	1 hr.	2 hrs.	3 hrs.	1 hr.	2 hrs.	3 hrs.	1 hr.	2 hrs.	3 hrs.
Apparent power density, g./cc.....	2.39	2.41	2.39	2.44	2.45	2.46	2.58	2.56	2.50	2.52	2.51	2.47	2.36	2.48	2.47	2.25	2.20	2.21
Sintered density, g./cc.....	6.33	6.34	6.29	6.52	6.59	6.50	6.83	6.83	6.70	6.80	6.80	6.78	6.68	6.60	6.51	6.30	6.21	6.09
Tensile strength, p.s.i.....	42,300	41,600	39,100	47,800	49,000	40,900	63,300	65,100	58,500	58,300	60,000	51,900	50,000	45,500	45,500	44,300	38,800	34,500
Elongation, percent.....	1.1	1.1	1.0	2.0	2.0	1.9	3.1	3.0	2.8	2.9	3.0	2.9	2.0	2.1	1.8	1.1	0.8	0.8
Linear shrinkage, percent.....	0.83	0.80	0.71	0.80	0.62	0.54	0.79	0.62	0.53	0.77	0.70	0.55	0.77	0.69	0.51	0.91	0.87	0.70
Hardness.....	B 42.4	B 43.1	B 40.7	B 52.7	B 53.1	B 49.2	B 63.3	B 64.9	B 59.2	B 61.0	B 61.9	B 58.8	B 60.1	B 61.2	B 58.1	B 54.2	B 56.2	B 49.1

Example 9.—Heat-treatment effect

The procedure of Example 8 is repeated, this time confining each heat treatment to a 60-minute period while

	Tensile strength, p.s.i.	Elongation, percent	Sintered density, g./cc.	Linear shrinkage, percent	Hardness
1% cobalt:					
Reduced.....	31,800	1.9	5.97	1.43	B 29.1
Heat-treated.....	55,200	4.0	6.79	0.72	B 59.7
1% nickel:					
Reduced.....	32,400	1.9	5.94	1.54	B 33.0
Heat-treated.....	65,100	2.3	6.77	0.90	B 70.9
1% molybdenum:					
Reduced.....	35,300	1.8	5.99	1.73	B 28.1
Heat-treated.....	65,500	1.9	6.90	0.69	B 74.5

more closely exploring the temperature range between 810° and 850° C., with temperatures controlled to $\pm 2^\circ$ C. Six test specimens are molded from each batch, with average test results as follows:

Temp., ° C.:	Tensile strength, p.s.i.	Elongation, percent	Linear shrinkage, percent	Hardness
810.....	58,500	2.0	0.73	B 60.2
820.....	58,800	2.0	0.75	B 59.4
830.....	60,700	2.9	0.69	B 62.0
835.....	63,300	3.1	0.79	B 63.3
840.....	63,800	3.0	0.82	B 64.1
845.....	58,600	2.1	0.84	B 60.0
850.....	49,100	2.0	0.80	B 52.7

The apparent powder densities range from 2.44 to 2.58 g./cc. and the sintered densities from 6.52 to 6.84 g./cc., for this series of tests.

Example 10.—Heat-treatment effect

The procedure of Examples 8 and 9 is repeated, this time subjecting a 7% copper alloy powder to heat treatment at 835° C. for periods of 30, 45 and 60 minutes, with the following results:

Heat treatment for (minutes).....	30	45	60
Compact: Green density, g./cc.....	5.91	6.40	6.65
Sintered Compact:			
Linear shrinkage, percent.....	1.21	0.97	0.79
Tensile strength, p.s.i.....	56,700	58,100	63,300

Example 11.—Reduction temperature and heat-treatment effects

Samples of each of the reduced powders prepared in Example 7 are heat-treated for one hour at 835° C. The heat-treated powders are then compacted and sintered as before, and subjected to physical testing, with results as follows:

	Reduction temperature					
	1,000° C.			1,125° C.		
	Batch			Batch		
	1	2	3	1	2	3
Heat treated powder:						
Apparent density, g./cc.....	2.44	2.12	2.10	2.69	2.58	2.57
Flow rate, sec./50 g.....	33.3	38.6	None	25.3	25.7	25.0
—325 mesh, percent.....	40.5	53.9	60.0	34.1	40.2	45.0
H ₂ wt. loss, percent ¹	0.51	0.52	0.67	0.30	0.22	0.20
Compact: green density, g./cc.....	6.48	6.37	6.30	6.65	6.73	6.66
Sintered compact:						
Sintered density, g./cc.....	6.68	6.55	6.52	6.83	6.80	6.76
Tensile strength, p.s.i.....	53,500	49,000	47,500	63,300	61,100	62,600
Elongation, percent.....	1.9	2.0	1.8	3.1	3.2	2.9
Linear shrinkage, percent.....	1.49	1.31	1.53	0.79	0.77	0.64
Hardness.....	B 60.9	B 59.4	B 63.0	B 63.3	B 58.9	B 60.4

¹ 1,100° C. for 30 minutes.

Example 12.—Effect of other metals

In accordance with the procedures of Examples 3 and 4, 7% copper alloy powders are prepared, each containing 1% cobalt, nickel or molybdenum. These are compacted

and sintered as in the previous examples, in both the as-reduced and as-heat-treated forms, with results as follows:

Example 13.—Use of elemental copper

The procedure of Example 1 is repeated, this time substituting for the cement copper an equivalent proportion (70 grams) of atomized copper powder finer than 100 mesh. The reduction is conducted at 1000° C. for 45 minutes, with heat treatment at 835° C. for one hour. After compacting and sintering as before, the reduced and heat-treated powders provide the following properties:

	Reduced	Heat-treated
Tensile strength, p.s.i.....	52,400	65,800
Elongation, percent.....	0.8	1.0
Sintered density, g./cc.....	6.39	6.70
Linear shrinkage, percent.....	0.85	0.09
Hardness.....	B 61.3	B 68.4

Example 14.—Other copper sources

The procedure of Example 1 is repeated, substituting for the cement copper equivalent quantities in proportion to their copper content of various other copper sources. After reduction at 1125° C. for 45 minutes and heat treatment at 825–845° C. for one hour, the powders are pressed and sintered as before to yield the following properties:

Copper source	Sintered density, g./cc.	Tensile strength, p.s.i.
Cupric oxide (83.02% Cu).....	6.81	50,100
Copper mill scale (88.64% Cu).....	6.80	51,200
Cement Copper No. 1 (90.80% Cu).....	6.85	63,300
Cement Copper No. 2 (93.04% Cu).....	6.88	68,000
Reduced copper powder (99.52% Cu)...	6.75	69,200

Example 15.—Effect of compacting pressure

A 7% copper alloy powder, prepared by reduction and heat treatment as described in Example 1, is combined with 0.75% stearic acid, compacted at various pressures,

and each compact is sintered at 1120° C. for 45 minutes in hydrogen. The physical properties, as a function of compacting pressure, are found to be as follows:

	Compacting pressure (t.s.i.)			
	30	40	50	60
Compact:				
Green density, g./cc.....	5.94	6.29	6.65	6.76
Green strength, p.s.i.....	2,058	2,501	3,591	4,829
Sintered compact:				
Tensile strength, p.s.i.....	43,400	52,900	63,300	75,200
Elongation, percent.....	2.9	3.1	3.0	1.8
Sintered density, g./cc.....	6.09	6.46	6.83	6.93
Linear shrinkage, percent.....	0.83	0.80	0.79	0.75
Hardness.....	B 46.0	B 61.3	B 63.3	B 69.2

Example 16.—Graphite effect with varying compacting pressures

Example 15 is repeated, this time incorporating 1% graphite in each heat-treated powder prior to compacting and sintering, with results as follows:

	Compacting pressure (t.s.i.)			
	30	40	50	60
Compact: Green density, g./cc.....	5.96	6.33	6.68	6.78
Sintered compact:				
Sintered density, g./cc.....	6.07	6.43	6.80	6.91
Tensile strength, p.s.i.....	72,700	91,500	109,100	137,000
Elongation, percent.....	1.9	2.0	2.0	0.9
Linear shrinkage, percent.....	0.86	0.84	0.81	0.80
Hardness.....	B 72.3	B 83.2	B 86.9	B 100.2

Example 17.—Coining effect

The procedure of Examples 15 and 16 are repeated, this time subjecting the final sintered piece to re-pressing and re-sintering under the same conditions used in the first pressing-sintering cycle. The properties achieved are summarized below:

	Compacting pressure (t.s.i.)			
	50	150	60	160
Compact: Green density, g./cc.....	6.65	6.63	6.76	6.74
Sintered compact:				
Sintered density, g./cc.....	6.83	6.79	6.92	6.90
Re-pressed density, g./cc.....	6.86	6.81	7.13	7.05
Re-sintered density, g./cc.....	6.93	6.85	7.20	7.11
Final tensile strength, p.s.i.....	77,200	111,300	82,900	123,600
Final elongation, percent.....	3.0	2.0	5.1	3.0
Final hardness.....	B 77.8	B 94.2	B 85.3	B 99.7
Linear shrinkage, percent:				
After 1st sintering.....	0.79	0.91	0.75	0.78
After 2nd compaction.....	0.08	0.01	0.06	0.01
After 2nd sintering.....	0.07	0.01	0.05	0.01

¹ 1% graphite additive.

Example 18.—Graphite effect with varying copper content

A series of ferrous alloy powders of varying copper content are prepared by the procedures of Examples 1 and 2, with reduction at 1125° C. for 45 minutes followed by heat treatment at 835° C. for one hour in hydrogen. Each powder is combined with 1% graphite. After pressing at 50 t.s.i. and sintering at 1120° C. for 45 minutes in hydrogen, physical properties are as follows:

	Copper content (percent)			
	4	7	14	20
Compact: Green density, g./cc.....	6.63	6.68	6.86	6.97
Sintered compact:				
Sintered density, g./cc.....	6.76	6.80	7.20	7.46
Tensile strength, p.s.i.....	103,700	109,100	112,000	112,900
Elongation, percent.....	2.1	2.0	2.0	1.9
Linear shrinkage, percent.....	0.77	0.81	1.46	1.96
Hardness.....	B 88.3	B 86.9	B 97.4	B 101.5

Example 19.—Post-sintering treatments

Sintered compacts prepared as in Example 18 are subjected to various additional treatments to further enhance physical properties, with results as follows:

	Copper content (percent)			
	14	17	214	220
Without graphite:				
Aged density, g./cc.....	6.76	6.78	7.33	7.59
Aged tensile strength, p.s.i.....	73,300	96,500	85,600	77,400
Elongation, percent.....	2.0	1.9	2.1	2.0
Linear shrinkage as aged, percent.....	0.76	0.48	1.65	2.47
Hardness.....	B 70.9	B 84.4	B 92.0	B 95.9
With 1% graphite:				
Aged density, g./cc.....	6.72	6.74	7.26	7.51
Aged tensile strength, p.s.i.....	118,100	133,300	147,000	137,200
Elongation, percent.....	1.9	1.9	1.9	2.0
Linear shrinkage as aged, percent.....	0.83	0.63	1.58	1.89
Hardness.....	B 85.4	B 94.4	B 110.8	B 109.4

¹ Quenched in water from sintering and aged at 485° C. in hydrogen for 30 minutes.

² Normalized in nitrogen at 1,000° C. for 60 minutes after sintering, then furnace-cooled to 370° C., held at 815° C. in hydrogen for 10-minute soak, quenched from this temperature in water, and finally aged at 260° C. one hour in air.

Example 20.—Effect of copper content in reduced powders

A series of ferrous alloy powders of varying copper content is prepared by the reduction procedure of Example 1A, with appropriate adjustment in the cement copper charged. The reduced powders are blended with 0.75% stearic acid lubricant, compacted at 50 t.s.i. and sintered at 1120° C. for 45 minutes in hydrogen. The physical properties are summarized in the table below:

	Percent						
	Percent		Sintered density, g./cc.	Tensile, ×10 ⁻³ p.s.i.	Percent		
	Copper	O ¹			Elonga- tion	Linear shrink- age	Hardness
50	2.....	1.22	5.90	37.8	1.9	1.82	B 27.2
	4.....	1.20	5.90	38.0	1.9	1.84	B 29.7
	6.....	1.26	5.92	38.5	1.1	1.81	B 35.2
	7.....	1.21	5.96	39.6	1.0	1.81	B 34.0
	8.....	1.20	5.98	39.7	1.0	1.80	B 35.5
	10.....	1.26	6.08	40.4	1.9	2.20	B 42.2
	12.....	1.23	6.29	45.9	1.8	2.35	B 49.3
	14.....	1.28	6.37	48.9	2.0	2.64	B 51.6
	16.....	1.26	6.54	51.6	2.1	3.17	B 55.6
	18.....	1.20	6.75	56.6	2.0	3.61	B 68.2
	20.....	1.97	6.87	60.5	3.0	3.68	B 69.6
	30 ²	1.20	6.81	79.7	3.9	8.13	B 90.9
	40 ³	1.84	6.88	58.2	3.8	8.48	B 86.4
	50 ⁴	1.99	7.27	42.0	4.0	8.44	B 53.8

¹ As indicated by percent weight loss in hydrogen at 1,100° C. for 30 minutes.

² Reduced at 1,050° C. for 30 minutes; sintered at 1,120° C. for 10 minutes.

³ Reduced at 950° C. for 30 minutes; sintered at 1,120° C. for 10 minutes.

⁴ Reduced at 950° C. for 30 minutes; sintered at 1,095° C. for 10 minutes.

Example 21.—Effect of copper content in heat-treated powders

Reduced powders prepared as described in Example 20 are heat-treated at 835° C. for 60 minutes in hydrogen, before compacting at 50 t.s.i. and sintering at 1120° C. for

45 minutes in hydrogen. The effect of the heat treatment on physical properties is summarized in the table below:

Percent		Sintered density, g./cc.	Percent Porosity	Tensile $\times 10^{-3}$ p.s.i.	Percent		
Copper	O ¹				Elonga- tion	Linear shrink- age	Hardness
2-----	0.29	6.80	13.8	49.0	4.9	0.77	B 42.2
4-----	0.30	6.82	13.8	51.6	4.8	0.80	B 50.0
6-----	0.27	6.83	14.0	53.3	3.9	0.75	B 53.9
7-----	0.30	6.83	14.1	63.3	3.1	0.79	B 63.3
8-----	0.23	6.89	13.4	68.9	4.0	0.93	B 66.1
10-----	0.28	6.89	13.6	82.8	3.9	1.14	B 77.2
12-----	0.31	7.02	12.3	83.7	4.1	1.32	B 80.0
14-----	0.30	7.12	11.2	84.8	4.0	1.55	B 80.9
16-----	0.28	7.23	10.1	85.9	4.0	1.97	B 83.1
18-----	0.23	7.38	8.4	86.7	3.9	2.32	B 84.8
20-----	0.23	7.46	7.8	87.4	4.1	2.58	B 88.1
30 ² -----	0.32	7.48	8.8	87.3	3.0	1.39	B 89.3
40 ³ -----	0.31	7.55	9.3	78.9	3.1	2.03	B 79.7
50 ⁴ -----	0.33	7.75	8.1	66.4	3.9	2.18	B 76.5

¹ As indicated by percent weight loss in hydrogen at 1,100° C. for 30 minutes.

² Reduced at 1,050° C. for 30 minutes; sintered at 1,120° C. for 10 minutes.

³ Reduced at 950° C. for 30 minutes; sintered at 1,120° C. for 10 minutes.

⁴ Reduced at 950° C. for 30 minutes; sintered at 1,095° C. for 10 minutes.

Example 22.—Copper-iron powder blends

For purposes of comparison, a series of copper alloys is prepared by prior art procedures, by blending appropriate proportions of 100 mesh reduced elemental copper and iron powders for 30 minutes, compacting at 50 t.s.i. and sintering at 1120° C. for 45 minutes in hydrogen. The properties obtained are summarized below:

Example 24.—Reduction temperature

	Grams
25 Iron mill scale -----	2376
Copper flue dust -----	288.7
Coke (Cabot) -----	303.8
Sugar (Sucrose) -----	38.1
Water -----	330.7

Percent copper	Sintered density, g./cc.	Percent porosity	Tensile, $\times 10^{-3}$ p.s.i.	Percent		
				Elonga- tion	Linear shrink- age ¹	Hardness
2-----	6.28	20.4	35.6	4.9	0.01	B 25.5
4-----	6.30	20.7	40.0	2.9	0.92	B 21.4
7-----	6.37	20.7	42.1	1.6	1.88	B 39.3
14-----	6.40	20.2	45.2	2.3	2.04	B 20.2
20-----	6.49	19.8	44.3	2.3	2.24	B 19.7
50 ² -----	7.02	16.6	26.6	4.5	0.99	B 3.2

¹ All samples expand on sintering.

² Sintered at 1,000° C. for 30 minutes.

Heat treatment of the powder blends by the procedure of Example 21 prior to compacting has no significant effect on the results.

Example 23.—Impact strength

The impact strengths of the novel alloys of Example 21 are compared with the values for conventional alloys prepared from blended copper-iron powders as in Example 22, and with iron compacts infiltrated with copper in the conventional manner, with results as follows:

Percent copper	Process	Impact strength, ft./lbs.		
		V-notch Charpy	Izod	Tension
7-----	Example 21-----	3.7	8.8	199.0
7 (+1% graphite)-----	do-----	4.8	8.0	186.0
7-----	Blended powders-----	3.5	3.8	86.8
7 (+1% graphite)-----	do-----	3.0	5.7	95.0
14-----	Example 21-----	4.6	-----	-----
15-----	Conventional infiltration-----	-----	3.2	-----
25-----	do-----	-----	5.0	-----
20-----	Example 21-----	5.3	-----	-----
30-----	do-----	5.1	-----	-----

The above ingredients are combined, milled into pellets, and reduced in 600 gram batches at 2000° F., 1850° F. and 1800° F. The gases evolved, containing carbon monoxide and carbon dioxide, are vented and burned. Thus, continuing combustion of vented gas provides an indication that the reduction is continuing. The reduction times required to approach "burn out" (completion of rection) are:

	° F.
55 1 hr. 48 min. -----	2000
2 hrs. 35 min. -----	1850
7 hrs. 20 min. -----	1800

The reduced cakes (12% copper) are crushed, ground and heat-treated at 1830° F. for one hour and then at 1535° F. for 30 minutes in dissociated ammonia. The annealed cakes are then crushed, ground and screened to -80 mesh.

The comparative powder properties are:

Reduction temp., ° F.	Apparent density, g./cc.	Flow rate, sec./50 g.	Percent			
			Carbon	H ₂ wt. loss	-325 mesh	Linear shrink- age ¹
2,000-----	3.11	20.7-----	0.03	0.55	28.8	2.12
1,850-----	1.87	No flow-----	0.39	1.11	80.7	6.18
1,800-----	1.92	do-----	0.60	1.64	86.3	6.05

¹ Pressing at 40 t.s.i. and sintering at 2,050° F. 35 minutes.

What is claimed is:

1. A process for preparing a particulate alloy comprising iron infiltrated with from about 1 to 50% by weight of copper, said process comprising the steps of mixing a reducible compound of iron with an appropriate proportion of a copper compound selected from the group consisting of elemental copper and reducible compounds of copper, heating said mixture under reducing conditions at a temperature between about 1010 (1850° F.) and 1150° C. (2100° F.), continuing said heating until said reducible compounds are substantially completely reduced to the metallic state having an iron-rich phase, subjecting said particulate alloy to heat treatment by maintaining said alloy for at least about 5 minutes at a temperature between the α - γ transition temperature of the iron-rich phase of said alloy and a temperature of about 150° C. below said transition temperature and thereafter cooling the alloy.
2. The process of claim 1 wherein the temperature during said reduction step is below the melting point of copper.
3. The process of claim 1 wherein the temperature during said reduction step is between about 1038 (1900° F.) and 1066° C. (1952° F.).
4. The process of claim 1 wherein said reducible mixture is characterized by a particle size finer than about 250 microns.
5. The process of claim 1 wherein said iron compound has a particle size finer than about 50 microns and said copper compound has a particle size finer than about 20 microns.
6. The process of claim 1 wherein said iron compound is an oxide of iron.
7. The process of claim 6 wherein said iron compound comprises hematite.
8. The process of claim 6 wherein said iron compound comprises magnetite.
9. The process of claim 6 wherein said iron compound is a reducible mill scale.
10. The process of claim 1 wherein said copper compound is an oxide of copper.
11. The process of claim 10 wherein said copper compound is cuprous oxide.

12. The process of claim 10 wherein said copper compound is copper flue dust.

13. The process of claim 1 wherein said copper compound is water-soluble and is introduced in the form of a water solution.

14. The process of claim 13 wherein said copper compound is cupric nitrate.

15. The process of claim 1 wherein said copper compound is elemental copper.

16. The process of claim 1 wherein said reducible mixture includes a minor proportion of a substance selected from the group consisting of nickel, cobalt, molybdenum, tungsten, and reducible compounds of said elements.

17. The process of claim 1 wherein said reducible mixture is blended with an aqueous dispersion of an organic adhesive prior to said reduction step.

18. The process of claim 1 wherein said reducing conditions are provided by incorporating finely divided carbon in said reducible mixture.

19. The process of claim 1 wherein said heating is conducted in a reducing atmosphere.

20. The process of claim 1 wherein said heat-treating temperature is less than 50° C. below said transition temperature.

21. The process of claim 1 wherein said heat-treatment is conducted at a temperature between about 825 and 845° C.

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