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[54] **OXIDE COATED FERROUS METAL POWDER**

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[51] **Int. Cl.** **B22f**

[58] **Field of Search** ..... 75/123 J, 125, .5 B, 75 AC, 75/75 BC

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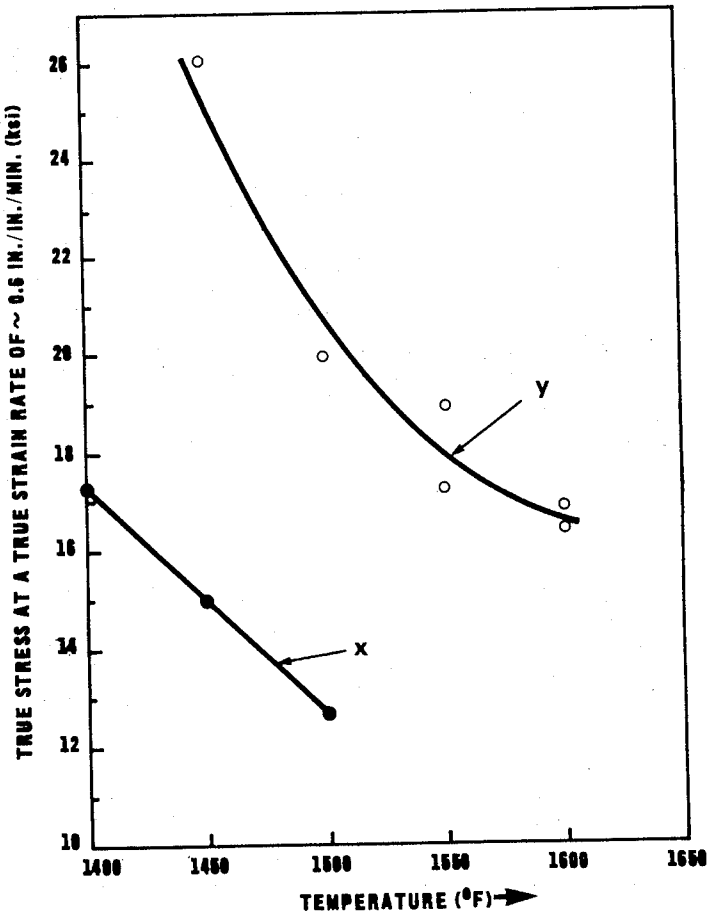
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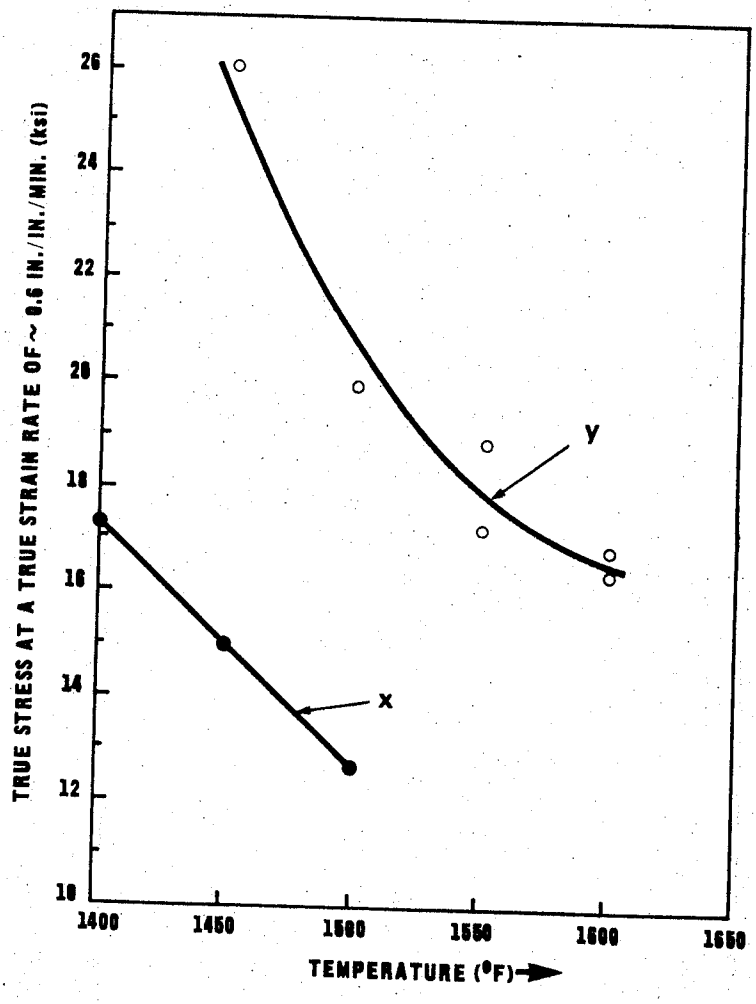
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[57] **ABSTRACT**

Steel powder forged at a temperature at which the steel is characterized by a microstructure containing specified percentages of ferrite and austenite contributes to low flow stress and other indicated advantages.

11 Claims, 1 Drawing Figure




TRUE STRESS AT A TRUE STRAIN RATE OF ~ 0.6 IN./IN./MIN. (ksi)
26
24
22
20
18
16
14
12
10
1400
1450
1500
1550
1600
1650
TEMPERATURE (°F)
y
x

OXIDE COATED FERROUS METAL POWDER

The subject invention is addressed in the main to powder metallurgy alloy steel forging.

As the metallurgist is aware, powder metallurgy has for some time played a prominent role in the production of a number of useful structural components. This has been particularly evident in respect of products not readily responsive to the more conventional melting-casting-working type processing, notably the production of intricately shaped products, certain refractory metals and alloys, various dispersion hardened materials, etc.

The virtues of powder metallurgy notwithstanding, the customary compacting and sintering techniques nonetheless gave rise to an attendant porosity problem, i.e., a problem in which products as finally processed are characterized by voids which in turn detract from various mechanical and/or physical properties, e.g., yield strength, impact energy, etc. This difficulty has quite naturally served to restrict the scope and application of powder technology.

To be sure, a number of techniques have been devised to minimize this porosity drawback. Mention might be made of repressing and/or infiltration as well as conducting both the compacting and sintering operations at high temperature. Such procedures, however, do introduce added cost and/or do not lend themselves to mass production techniques.

In recent years powder metallurgy (P/M) forging, an historically old and near forgotten art, has received considerable attention since it offers an economically attractive panacea for virtually eliminating porosity while being amenable to automation. In large part, at least to date, powder forging investigations in the steel area seemingly have been confined to relatively conventional wrought alloy compositions and those previously used in the sintering industry, perhaps because their properties were of a known quantity and thus offered some basis for comparison. Illustrative of this are the higher carbon AISI 4600 type steels.

Be that as it may, the present invention is particularly concerned with new steels which by virtue of their chemistry enhance the carrying forth of the P/M forging process and/or which by virtue of the forging process are rendered particularly useful. It is to be understood, however, that the instant invention does not exclude steels the composition per se of which might heretofore have been known.

It has now been discovered that improvements involving alloy steel powder forging can be brought about provided the forging operation is controlled such that it is conducted at a temperature at which the steel is characterized by a special structure as herein described.

Generally speaking, the present invention contemplates the forging of powder alloy steels characterized at the temperature of forging by a microstructure in which the alloy steel powders are comprised essentially of the ferrite and austenite phases, these phases mutually coacting such that grain growth is retarded during recrystallization. Advantageously, at the forging temperature both the ferrite and austenite are present in a volume percentage of at least about 10 percent with their respective grains not exceeding an average ASTM grain size of about 10, the grain size most advantageously being not greater than about ASTM 12.

In accordance herewith, upon bringing the steels to the desired microstructural condition and forging at a temperature which does not deleteriously disrupt this condition, it is deemed that any one or more of a number of benefits follow, e.g., improved die filling, lower forging loads for a given configuration, less oxidation, the likelihood of decreased die wear, breakage or distortion, lower cost, etc.

For example, with respect to the low alloy steels herein illustrated, it was found that such steels when in the proper ferritic — austenitic condition manifest a markedly lower flow stress (less resistance to flow) than might otherwise be the case. In conjunction therewith it was further determined that a significantly lower forging temperature could be used as opposed to the necessity of using one much higher as is the prevailing conventional practice. By reason of such (and other) characteristics, it is considered that, for example, longer die life should obtain and, thus, less downtime as well. And as is known, given the complexities of die configurations coupled with the production problems associated therewith, die life is an important economic component in the equation determinative of whether the manufacture of a given product is likely to be competitive.

The following novel alloy steel compositions are considered particularly beneficial since in addition to manifesting low flow strengths at the prerequisite forging temperature they afford high strength at room temperature: about 0.05 to 0.15 percent carbon; about 0.8 to 2.5 percent silicon; up to about 2 percent manganese; about 0.5 to 4 percent nickel; about 0.2 to 2 percent molybdenum; up to about 2 percent, e.g., 0.8 to 1.75 percent, copper; up to 0.2 percent, e.g., 0.02 to 0.12 percent, columbium; up to 0.15 or 0.25 percent, e.g., from 0.01 or 0.03 to 0.12 percent, oxygen, the balance being essentially iron and impurities. Such compositions can be compacted and forged to virtually 100 percent density at about 1400°–1500°F., e.g., 1450°F., in contrast to temperatures on the order of 1650°–1800°F. commonly employed in conjunction with current low alloy steel P/M forging practice.

In terms of the ferrite and austenite phases, while the volume percentage of each may be as low as 4 or 5 percent, in striving for an overall good combination of physical and/or mechanical properties, including flow strength, room temperature strength, etc., at least 10 percent, and most beneficially about 20 percent or more, of each should be present at the forging temperature. A microstructure at that point containing, say, 1 or 2 percent ferrite, balance austenite would be rather unsuitable since, inter alia, it would exhibit a higher flow strength and there would be insufficient ferrite to prevent undesirable grain growth in the austenite. In this connection and as indicated above, the grains of these phases prior to forging and at the temperature of forging should be at least about ASTM grain size 10 on average. The grains can be larger, e.g., ASTM 8, but generally at the expense of some mechanical or physical property. ASTM 14 or smaller is considered beneficial particularly in respect of various properties. Grain size applies to the forged products as well.

Concerning the powder particles, while elemental powders might be blended and sintered to the desired composition, it is deemed much preferable to use prealloyed powder. As will be appreciated by the artisan, this can, for example, be accomplished through atom-

ization in which a liquid melt of desired composition is formed and directly converted to powder by using air, steam, inert gas, vacuum or water to bring about atomization. Water atomization, with or without an inert gaseous stream, is considered appropriate since it is commonly employed and is relatively inexpensive. Prealloying and atomization provide for small particle size and grain size. Moreover, it is preferred that the particles as alloyed be of irregular shape as opposed to, say, spherical. This enhances particle interlocking.

The alloy powders should not exceed about 500 microns (including oxide film), preferably being less than 275 microns, an advantageous powder mix being one containing not more than 25 percent of powder less than 40 microns in size, the remainder being up to 225 or 275 microns. The surface of the powder particles is substantially, if not completely, comprised of an oxide film but this is beneficial in accordance herewith. This inhibits grain growth within each particle as well as inhibiting growth across interparticle boundaries. Though up to possibly 1 percent oxygen can be present in the steels, high oxygen impairs toughness, detracts from compacting and sintering prior to forging and retards interparticle welding during forging. The oxygen level should not exceed about 0.25 percent and the oxide film thickness should be less than about 5 or 10 microns.

The prealloyed powder particles are thereafter compacted to a preform, the shape of which will often, though not necessarily, depend on the shape of the final product. Thereupon, the prealloyed preform is heated to obtain the desired ferritic-austenitic microstructure whereupon it is forged to shape and full or nearly full density. As is often done in practice, an appropriate lubricant can be added to the prealloy powder before pressing to the preform. Also, the preform can be heated (sintered) prior to forging in accordance with usual practice. Subsequently, the product may, if desired and depending on composition, be further processed, e.g., machined or aged in the case of age hardenable materials or normalized, carburized, nitrided, etc.

In order to give those skilled in the art a better appreciation of the advantages of the invention, the following data are given.

Several steel compositions were prepared by atomizing prealloyed melts, the chemistry of which are given in Table I. The atomized powders were made from air induction melted heats using a magnesia crucible. Electrolytic iron and nickel were first charged and melted and then molybdenum pellets were added. The melts were heated to 2850°F. and pig iron (to add carbon for carbon boil) added. Following a 5 minute boil half the silicon (ferro form) was added together with pig iron and, if any, ferromanganese, ferrocolumbium and copper, the remainder of the silicon being added. The melts were tapped at 2850°F. atomized with argon at 400 psi under an argon atmosphere and the prealloyed powders were then screened into various mesh sizes. Argon was used since water atomization facilities were unavailable.

TABLE I

Alloy	C	Si	Ni	Mo	Cu	Cb	O
1	0.069	1.22	1	0.24	1.4	0.015	0.06
2	0.11	2.38	2.6	0.93	1.0	0.071	0.04
3	0.11	2.44	2.5	0.89	n.a.	n.a.	0.05
4	0.072	1.25	0.98	0.28	n.a.	n.a.	0.058
5	0.085	1.19	0.96	0.58	n.a.	0.02	0.07
6	0.084	1.28	0.98	0.50	1.6	n.a.	0.05
7	0.12	2.35	2.8	1.8	n.a.	0.037	0.058
8	0.10	2.39	2.8	1.7	1.4	n.a.	0.048
9	0.10	2.47	3.1	1.8	1.5	0.02	0.05
10*	0.14	1.64	1.03	0.35	n.a.	n.a.	n.d.

* contained 0.12% Ti and 0.002% B and 0.65% Mn
n.a. = not added

While all the alloys above were tested, a few specific examples will be given as illustrative.

EXAMPLE I

This example is illustrative of a low alloy steel combining high yield strength and good ductility, it being a specific objective of the invention to provide a steel with a yield strength of at least 90,000 psi together with a tensile ductility of about 10 percent or more.

Using −40 to +100 mesh particles, Alloy 1 was sealed in a mild steel metal can (3 inches inside diameter, 6 inches long, ¼ inch thick walls) and rolled (starting temperature of 1650°F.) to fully dense (no obvious voids) ⅜ inch thick plate, the mill speed being about 40 ft./min. The can was machined off and specimens for room temperature and hot tensile tests were cut from the plates, aged about 4 hrs. at 1000°F. and air cooled. The following tensile properties were obtained at room temperature: yield strength, 125,100 psi; ultimate tensile strength, 125,100 psi; elongation (in 0.55 inch), 11.5 percent; reduction in area, 30.5 percent; R_c hardness, 27.2.

To simulate expected powder forging behavior, the steel was tensile tested at various true strain rates. Flow strength was determined at the various strain rates at 1450°F. The data include a similarly processed and currently used powder forging steel (Alloy A) nominally containing about 0.5% C., .35% Mn., 0.01% P, 0.02% S, 0.45% Ni and 0.55% Mo.

True Stress (psi)		True Strain in/in/min.
Alloy 1	Alloy A	
4,570	14,200	0.006
9,160	16,930	0.062
14,980	26,050	0.606

It will be observed that at the temperature of 1450°F. Alloy 1 of the invention had a markedly lower flow strength at all three strain rates. This should contribute to enhanced die filling and/or result in less die wear and distortion in hot forging. It should be emphasized this

was achieved notwithstanding that the yield strength thereof exceeded that of Alloy A by about 36,000 psi, the elongation of the latter being only 7.5 percent. Actually, Alloy 1 had a lower flow stress at 1450°F. than Alloy A at 1650°F. Alloy 1 upon being can hot-rolled, heated at 1450°F. for ½ hour and cooled (approximating an air cool) had a fine grained microstructure which by visual observation consisted of about 10 to 20 percent ferrite, less than 2 percent austenite (as determined by X-ray), the balance being the decomposition products of austenite. Grain size was less than ASTM 12.

EXAMPLE II

Alloy 2 is illustrative of a moderately higher strength steel than Alloy 1 but with less ductility. It was processed in the same manner as Alloy 1 and displayed a yield point of 130,700 psi, an ultimate tensile strength of 149,000 psi, an elongation (in 0.55 inch) of 5.5 percent, a reduction in area of 15 percent and a hardness of R_c 33.5. Its flow strength at 1450°F. is given below together with the commercially produced Alloy A of Example I.

True Stress (psi)		True Strain in/in/min.
Alloy 2	Alloy A	
5,740	14,200	0.006
10,570	16,930	0.064
18,290	26,050	0.528

Alloy 2, tested as in Example I, had a microstructure of about 25 – 35 percent ferrite the remainder being similar to Alloy 1. Grain size was about ASTM 12–14.

EXAMPLE III

Alloy 3 is representative of a steel useful for carburization purposes subsequent to forging. After applying a heat treatment cycle quite similar to that used in carburizing (but without the carbon), the heat treatment consisting of 40 min. at 1700°F., oil quenching, plus 3 hrs. at 300°F. and air cooling, the following properties were obtained: yield strength (0.2 percent offset), 140,300 psi; ultimate tensile strength 188,600 psi; elongation (in 0.55 inch), 14.5 percent; and a reduction in area of 33 percent. These properties are indicative of those to be expected in the core of a carburized part. Carburizing at 1700°F. at 0.95 percent carbon potential, then oil quenching and tempering at 300°F. for 3 hours resulted in lower ductility in another specimen of Alloy 3. About 15 – 25 percent ferrite, approximately 2 percent austenite with the balance being the decomposition products of austenite, constituted the microstructure as tested in Example I.

FIG. 1 graphically depicts the true stress at a true strain rate of about 0.6 in/in/min at a temperature of 1450°F. for Alloys 1–9. Curve X is specific to Alloy 1 and spans the 1400°F. – 1500°F. temperature range. Curve Y is specific to Alloy A. It can be seen that 1400°F. stress for Alloy 1 is comparable to that of Alloy A at 1600°F. despite the fact that the latter had the benefit of the much higher temperature.

Among other attributes of the above steels in comparison with the high carbon AISI 4600 steels and other proposed high carbon steels, might be listed the following: (*a*) they do not require heating to the austenite range, e.g., 1700°F. and above, followed by rapid cooling and tempering, a treatment which leads to unwanted oxidation (absent special processing) and which can contribute to distortion; (*b*) they do not require 0.4 – 0.5 percent carbon to provide adequate strength upon hardening. High carbon does render such prior art steels austenitic at the forging temperatures employed and does result in high strength steels, but, high carbon also can contribute to die wear and distortion. The present invention minimizes this and yet is capable of delivering yield strengths up to 150,000 psi, if desired; and (*c*) they do not require carefully controlled atmospheres during sintering and heating for forging to prevent carburization.

It perhaps should be also mentioned that conventional pre-conditional treatments are unnecessary as a prerequisite to obtaining a fine grain. In prior art wrought alloy steel processing it is common, for example, to hot work a steel while cooling down to and into a duplex region. It is the pre-conditioning working operation which is largely responsible for a fine grain; otherwise, a coarse grain results. Forging as contemplated herein involves virtually an isothermal operation during which continuous recrystallization takes place. This contributes to maintaining a fine grain structure but without need of preconditioning.

With regard to the roles of the individual constituents of the steels of specific composition given herein, nickel contributes to strength, hardenability and broadens the temperature range over which stable ferrite-austenite structures can be obtained. But, an excess is unwarranted due to cost and may increase resistance to flow at 1400°F. to 1500°F. by unnecessarily increasing the amount of austenite. Moreover, nickel is particularly useful in bringing about the proper microstructure particularly in counterbalancing the significant effect of silicon in contributing to the formation of ferrite. While manganese might be used in lieu of nickel, toughness suffers and manganese tends to give an oxidation problem. It should be held to less than 0.4 percent.

The molybdenum need not exceed 1.2 percent. At nickel percentages of 1 and 3 percent the higher molybdenum levels appear to detract from tensile strength. A range of 0.2 to 0.6 percent is satisfactory. Columbium tends to increase flow stress and preferably should not exceed about 0.1 percent. It does contribute to tensile strength and a range of 0.02 to 0.08 percent is of benefit. Silicon, as noted, promotes the formation of ferrite but does lower toughness at the higher levels. It is to advantage that it not exceed 2 percent. As to carbon, while it can be omitted it does confer strengthening qualities and it is preferred that at least 0.02 or 0.04 percent be present. High levels, e.g., 0.4 or 0.5 percent, are unnecessary and although they could be used in a properly balanced structure, at desired forging temperatures could result in virtually completely austenitic steels such that, other factors remaining equal, excessive die wear or distortion could ensue. Copper provides age hardening and strength.

Other elements such as chromium, aluminum, vanadium, titanium, tungsten and cobalt can be present. These constituents need not exceed 2 or 3 percent each, with the aluminum plus titanium preferably not exceeding 0.5 percent. If employed, attention must be given to their respective ferrite, austenite forming tendencies. Aluminum and tungsten might be used in lieu

of or together with lower levels of silicon for the purpose of forming ferrite. However, silicon is much preferred since tungsten is of high density and is expensive and an equivalent percentage of aluminum would tend to impart an oxidation problem.

Where a combination of low flow stress over the temperature range of about 1400°F. – 1500°F. coupled with a room temperature yield strength of 80,000 – 120,000 psi and good tensile ductility (above 7.5 percent) is desired, the following range of composition is considered most advantageous: about 0.05 – 0.15 percent carbon, about 0.8 to 1.5 percent silicon, about 0.5 to 1.2 percent nickel, about 0.2 to 0.4 percent molybdenum, about 1 to 2 percent copper, up to 0.1 percent columbium and the balance iron. While this composition could be carburized, the usefulness of copper, which imparts age hardening capability, would be largely lost since an aging treatment at 900° – 1000°F. could not be used to advantage on a high carbon martensitic part. Thus, for carburizing, these steels should be copper-free or contain less than about 0.5 percent. For higher yield strengths, e.g., 130,000 psi and above, the silicon, nickel and molybdenum levels should be about 2 to 2.5 percent, 2 to 3 percent and 0.8 to 1.2 percent, respectively.

While the steels of specific composition described herein have been directed to powder metallurgy forging, they are also useful in the production of wrought and cast products such as rod, plate and sheet (mill products) particularly when characterized by a fine-grained microstructure.

While the present invention is useful in the production of a variety of forged products it is deemed particularly applicable in forming such components as pinions, connecting rods, gears and side gear blanks.

Although as a practical matter it is unlikely, other phases in addition to ferrite and austenite can be present at the forging temperature in amounts which do not have an adverse affect. Inclusions, e.g., sulphide and silicate inclusions, are not deemed to be phases as contemplated herein but can be present in small quantities. Upon cooling from forging, the microstructures are comprised of ferrite and transformed products of austenite, e.g., martensite, bainite, etc. This will depend on composition and rate of cooling as will be appreciated by the artisan. Also, a small percentage of austenite, say, up to 5 percent, might also be present.

Although the present invention has been described in conjunction with preferred embodiments, it is to be understood that modifications and variations may be resorted to without departing from the spirit and scope of the invention as those skilled in the art will readily understand. For example, in addition to powder forging, the alloy powders can be extruded or otherwise worked. Such modifications and variations are considered to be within the purview and scope of the invention and appended claims.

I claim:

1. Steel powder particularly adapted for use in powder forging and formed of a composition consisting of about 0.05 to 0.15 percent carbon, about 0.8 to 2.5 percent silicon, up to less than 1 percent manganese, about 0.5 to 4 percent nickel, about 0.2 to 2 percent molybdenum, up to about 0.2 percent columbium, up to 2 percent copper, about 0.01 to 0.25 percent oxygen, the balance being essentially iron, the particles forming the powder being of irregular shape and further characterized by a particle size of not greater than about 500 microns, a grain size not less than an average ASTM grain size of about 10 and with their outer surfaces being substantially enveloped by an oxide film not greater than about 10 microns in thickness.

2. Steel powder in accordance with claim **1** in which the oxygen content does not exceed about 0.15 percent, the particle size is less than 275 microns, the oxide file is not greater than about 5 microns, manganese, if any, is not greater than 0.4 percent, the powder also being virtually columbium free.

3. Steel powder in accordance with claim **2** containing copper in an amount sufficient to impart significant hardening and strength upon being cooled from an aging treatment of 900°F. – 1000°F.

4. Steel powder in accordance with claim **3** which contains at least 1 percent copper, at least 0.01 percent columbium and in which the molybdenum does not exceed 1.2 percent.

5. Steel powder in accordance with claim **2** containing from 0.8 to 1.5 percent silicon, up to 0.4 percent manganese, about 0.5 to 1.2 percent nickel and about 0.2 to 0.5 percent molybdenum.

6. Steel powder in accordance with claim **5** which contains from about 1 to 2 percent copper.

7. Steel powder in accordance with claim **1** containing from 2 to 2.5 percent silicon, about 2 to 3 percent nickel, about 0.8 to 1.2 percent molybdenum and about 1 to 2 percent copper.

8. A wrought or cast alloy steel having a chemical composition as set forth in claim **1.**

9. Steel powder particularly adapted for use in powder forging and formed of a composition consisting of up to 0.4 percent carbon, about 0.8 to 2.5 percent silicon, up to less than 0.4 percent manganese, about 0.5 to 4 percent nickel, about 0.2 to 2 percent molybdenum, up to 0.2 percent columbium, up to 2 percent copper, oxygen present in an amount not greater than 1 percent, up to 3 percent each of cobalt, chromium, tungsten, vanadium, up to 0.5 percent in total of aluminum plus titanium, and the balance essentially iron.

10. A powder forged connecting rod formed of a composition as set forth in claim **1.**

11. A powder forged gear formed of a composition as set forth in claim **9.**

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