March 17, 1959 M. F. BECHTOLD 2,878,113

Fe-Si-Ti COMPOSITION OR PRODUCT AND PROCESS OF PREPARING SAME

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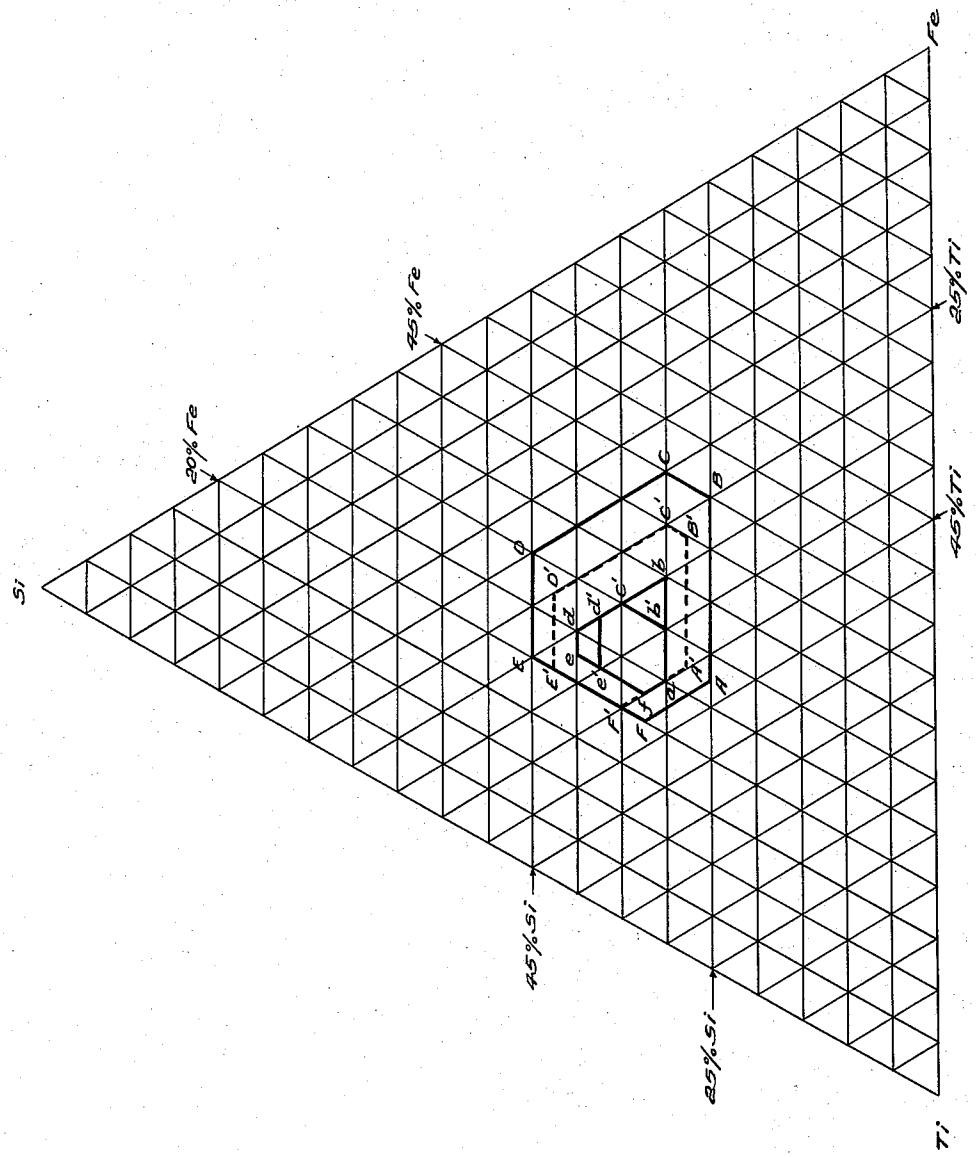

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Fe-Si-Ti COMPOSITION OR PRODUCT AND PROCESS OF PREPARING SAME

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This invention relates to powder metallurgy compositions and to shaped objects of alloys having high heat and oxidation resistance. More particularly, this invention relates to new iron-silicon-titanium powder compositions, to shaped objects of iron-silicon-titanium alloys that are hard, strong, creep-resistant and resistant to oxidation at high temperature, and to methods for preparing such shaped objects.

Considerable effort has been expended in the search for metals or alloys that are resistant to attack by acids and alkalies and that can be subjected to high temperatures in air without deleterious effects, such as loss of strength, warpage or oxidation. Although metallic compositions showing some of these advantageous properties have been made, they are usually difficult to obtain in the form of shaped objects, are usually quite expensive, and domestic sources for the metals involved are often not adequate for normal needs. In addition to the need for strong metallic structures having high heat and oxidation resistance, low density compositions resistant to abrasion are also desired.

It is an object of this invention to provide novel powder metallurgy compositions. A further object is to provide novel iron-silicon-titanium powder compositions. A still further object is to provide shaped objects of iron-silicon-titanium alloys that are hard, strong, creep-resistant and resistant to oxidation at high temperatures. Another object is to provide a novel method for preparing the aforesaid shaped objects. Still another object is to provide shaped articles of iron-silicon-titanium metallic structures of low density and which are resistant to abrasion in addition to having high heat and oxidation resistance. Other objects will appear hereinafter.

These and other objects of this invention are accomplished by providing a powder metallurgy composition consisting of a powder having substantially all particles, i. e., at least 95% (by weight), less than 75 microns in size and consisting essentially of iron, silicon and titanium in the proportions of 20–45% iron, 25–45% silicon and 25–47.5% titanium. Preferred compositions are those having an average particle size below about 60 microns with at least 25% (by weight) of the particles being less than 45 microns in size.

The drawing is a three-component diagram of

Fe—Si—Ti

alloy compositions in weight percent.

The area within the polygon ABCDEF embraces the novel Fe—Si—Ti alloy compositions of this invention consisting essentially of 20–45% Fe, 25–45% Si, and 25–47.5% Ti.

The area within the polygon A'B'C'D'E'F' embraces the preferred alloy compositions of this invention consisting essentially of 20–40% Fe, 27.5–42.5% Si, and 30–45% Ti.

The area within the polygon *abcdef* embraces particularly preferred alloy compositions of this invention consisting essentially of 22.5–35% Fe, 30–40% Si, and 35–45% Ti.

The area within the polygon *ab'c'd'e'f* embraces the most preferred alloy compositions of this invention consisting essentially of 22.5–30% Fe, 30–37.5% Si and 35–45% Ti.

Strong metallic objects resistant to oxidation, high temperatures and abrasion are prepared by pressing these powders in the form of the desired object and then heating rapidly to a temperature of at least 900° C., or by hot-pressing. Such objects are also part of this invention. For increased oxidation resistance and high temperature strength, compositions containing 20–40% Fe, 27.5–42.5% Si and 30–45% Ti are preferred, while compositions in the range of 22.5–35% Fe, 30–40% Si and 35–45% Ti possess these properties to a still greater extent. Compositions with maximum melting point and other desirable properties contain 22.5–30% Fe, 30–37.5% Si and 35–45% Ti, and include, for example, compositions of the following atom ratios:

1Fe—3Si—2Ti; 2Fe—5Si—4Ti; 2Fe—4Si—3Ti

and 2Fe—5Si—3Ti. The X-ray diffraction patterns of these most preferred alloy compositions, after heating rapidly to a temperature of at least 900° C. or hot pressing, show them to be substantially free of the elements Fe, Si, Ti, their known binary compounds and their reaction products with the atmosphere. These alloys are thought to be composed of new ternary metal silicide compounds, such as $FeSi_3Ti_2$.

Particularly useful compositions for the production of shaped objects are obtained by grinding together ferrotitanium and silicotitanium in such proportions that the resulting powder falls within the ranges of composition and particle size described above. Such compositions are readily shaped by cold-pressing and converted to alloy objects by heating to a temperature of 1100–1400° C. within a short period of time, e. g., a few seconds to 10 minutes. Alloy objects are also obtained by hot-pressing the powders at 2000–6000 p. s. i. and 1100–1400° C. for 1 to 30 minutes or more.

When compositions outside the above-defined ranges are used, the final products contain appreciable amounts of uncombined elements and/or are deficient in such properties as strength, hardness, resistance to high temperature, resistance to oxidation and freedom from mechanical defects. For example, larger proportions of Fe, although desirable because of its low cost, lead to softer, magnet-responsive alloys poor in oxidation resistance and in high temperature strength. Increasing proportions of Ti likewise lead to softer, less refractory alloys, while increasing proportions of Si result in more brittle, lower melting products.

The following examples illustrate the preparation and properties of the compositions and derived shaped objects of this invention.

EXAMPLE I

Coarse chunks (35.8 g.) of a commercial ferrosilicon alloy (22.02% Si, the remainder being substantially all

Fe) were ball-milled in a one-quart porcelain ball mill for 66 hours with 52 g. of $TiSi_2$ powder to yield a mixed fine powder with input ratio (Fe–Si–Ti) of 31.80–40.92–27.28 by weight. Approximately 2 g. of this powder was pressed at 40 tons per square inch (t. s. i.) pressure in a cylindrical steel die to yield a "green" pellet. This pellet was placed for two minutes inside a muffle furnace operating in air at 1360° C. After removal from the furnace and cooling, the pellet was found to be strong, metallic in appearance, harder than glass and substantially unoxidized on the surface. It was not responsive to a magnet, indicating that reaction had occurred during firing, since the input ferrosilicon and the unfired pellet were responsive to a magnet.

EXAMPLE II

The ball-milled powder of Example I was pressed at 13 t. s. i. in a rectangular steel die (nominal size 1½" x ½" with rounded corners) to yield a "green" bar, which was fired for 2 minutes in air at 1360° C. The weight and dimensions before and after firing were:

	Weight (g.)	Thickness (cm.)	Width (cm.)	Length (cm.)	Bulk Density (g./cc.)
Green	10.466	0.6935	1.2374	3.85	3.23
Fired	10.570	0.6372	1.1648	3.67	3.96

The resultant porous, hard, metallic bar withstood dropping on a hard floor without fracture and when heated successively at 1000°, 1100°, 1200° and 1300° C. in air (16 hours at each temperature with cooling and weighing before and after each heat) stabilized in weight at 1100° C. The total weight gain during the 1200° and 1300° C. heats was only 0.45%.

EXAMPLE III

Reduced iron powder (7.8 g.) was ball-milled for 48 hours in a porcelain ball mill with $TiSi_2$ powder (10.4 g.) to yield a fine mixed powder with input ratio (Fe–Si–Ti) of 42.86–30.82–26.32. A "green" bar was prepared and fired 2 minutes in air at 1300° C. It attained 1300° C. within 15 seconds and exceeded this temperature within 18 seconds (exhibiting "calorescence"). The following results show weight and dimensional changes on firing:

	Weight (g.)	Thickness (cm.)	Width (cm.)	Length (cm.)
Green	9.891	0.6500	1.2374	3.85
Fired	9.931	0.5682		3.42

A hard metallic bar was obtained by the firing. A section of this bar was weighed and measured before and after heating at a series of temperatures with results as follows:

	Initial	After 16 hrs. successively at—		
		1,000° C.	1,100° C.	1,200° C.
Weight (g.)	2.2632	2.2675	2.2716	2.2819
Thickness (cm.)	0.6380	0.6392	0.6402	0.6455
Width (cm.)	0.5703	0.5718	0.5718	0.5757
Length (cm.)	1.3500	1.3588	1.3627	1.3658

The sample was essentially all hard metal at the end of the heat treatments.

EXAMPLE IV

Reduced iron powder (67 g.) and silicotitanium alloy, (98.3 g.) (analysis: Fe, 1.05%; Si, 44.83%; Ti, 45.10%; others, 9.02%) about 20 mesh in size were ball-milled for 22 hours in a porcelain ball mill to yield a fine powder having an input ratio (Fe–Si–Ti) of 43.49–28.17–28.34. A 25 g. sample of this powder was mixed with 4.9 g. of 15% aqueous NaOH, and dried. A green bar prepared from this powder was fired as in Example III. It attained 1300° C. in 19 seconds and reached a maximum temperature higher than 1300° C. in 22 seconds. The product was hard, strong and metallic. The following table shows weight and dimensional changes:

	Weight (g.)	Thickness (cm.)	Width (cm.)	Length (cm.)
Green	11.196	0.7178	1.2437	3.85
Fired	11.261	0.6517	1.1690	3.29

A section of this bar was heat-treated in air as in Example III with the following results:

	Initial	After 16 hrs. successively at—			
		1,000° C.	1,100° C.	1,200° C.	1,300° C.
Weight (g.)	2.1801	2.1990	2.1974	2.1968	2.1998
Thickness (cm.)	0.6422	0.6598	0.6563	0.6542	0.6548
Width (cm.)	0.6455	0.6612	0.6593	0.6597	0.6637
Length (cm.)	1.1960	1.1945	1.2050	1.2000	1.2000

After the initial heat at 1000° C., the bar showed substantial stability in weight and dimensions and at the end of the test was very hard and metallic.

EXAMPLE V

Ferrotitanium alloy lumps (19.4 g., analysis: Fe, 65.7%; Si, 4.62%; Ti, 28.44%, Al, 1.21%; and C, 0.04%) were ball-milled with the silicotitanium alloy (35.6 g.) of Example IV to yield a fine powder (input ratio: 25.44 Fe–32.70 Si–41.86 Ti). A bar was pressed and fired as in Example III. It attained 1300° C. within 24 seconds and a maximum temperature above 1300° C. in 31 seconds. After cooling, the bar was hard, porous, sound, metallic and substantially undistorted. It survived repeated drops of 12 feet to a hard floor without fracture. The following weight and dimensional changes occurred during firing:

	Weight (g.)	Thickness (cm.)	Width (cm.)	Length (cm.)	Bulk Density (g./cc.)
Green bar	11.933	0.7933	1.2374	3.85	3.22
Fired bar	12.113	0.7867	1.2218	3.80	3.38

The following changes occurred on successive heat-treatments in air of a section of this bar:

	Initial	After 16 hrs. successively at—			
		1,000° C.	1,100° C.	1,200° C.	1,300° C.
Weight (g.)	2.2756	2.3641	2.3609	2.3596	2.3609
Thickness (cm.)	0.6388	0.6450	0.6424	0.6438	0.6445
Width (cm.)	0.7702	0.7810	0.7700	0.7715	0.7765
Length (cm.)	1.4442	1.4472	1.4500	1.4500	1.4500

Thus, the alloy showed considerable stability after the 1000° C. heat.

EXAMPLE VI

Titanium disilicide powder (90 g.) and steel powder (45 g.) (analysis: Fe, 96.37; Si, 0.21; others, 3.42) were ball-milled to yield a fine mixed powder (input ratio: 32.49 Fe–36.46 Si–31.05 Ti). Two portions of this powder were converted to hard metallic bars by (*a*) pressing at 9 t. s. i. and then firing as in Example III, and (*b*) hot-pressing at 1300° C. under 3000 p. s. i. for 10 minutes in a graphite mold. In (*a*), 1300° C. was reached within 30 seconds and a maximum temperature above

this in 38 seconds. The following table shows comparative data on these objects:

Test	Bulk Density (g./cc.)	Porosity (percent)	Hardness (Knoop) at Loads of—		
			10 g.	100 g.	1,000 g.
(a)	4.29	10.5	1,895	879	722
(b)	4.78	0.63	1,948	1,222	1,052

The effect of heat-treatment in air on these objects is shown in the following table:

After 16 hours successively at—	Changes in Object (a)[1]		Changes in Object (b)[1]	
	Weight (percent)	Dimension [2] (percent)	Weight (percent)	Dimension [2] (percent)
1,000° C.	+0.54	+0.02	+0.17	+0.38
1,100° C.	+0.62	+0.16	+0.31	+0.79
1,200° C.	+0.68	+0.32	+0.52	+1.53
1,300° C.	+0.76	+0.14	+1.48	+4.02

[1] Total accumulated growth based on initial size and weight.
[2] Average of three dimensions.

EXAMPLE VII

In the following tests, the ingredients (Table A) were mixed by dry ball milling (about 40 hours) until adequate particle size reduction was obtained. A typical screen analysis after ball milling was that shown in Test No. 5 as follows:

Screen Size Mesh	Particle Size μ=Microns	By weight, Percent
>60	>250	1.88
60-80	250-177	0.67
80-100	177-149	0.09
100-150	149-99	0.31
150-200	99-74	0.35
200-325	74-44	43.93
<325	<44	52.77

The resultant powders were placed in a graphite mold and a pressure of about one-half that eventually to be used (Table B) was applied. The mold was heated by induction to the temperature indicated in Table B. This required 24 to 89 minutes, depending on the size of the mold and molded object. The final pressure was applied as the assembly attained about 1100° C. The pressure and temperature indicated were held for 2 to 10 minutes, depending on the progress of consolidation as shown by diminishing volume of the molded object. The pressure was then released and the hot-pressed bars were cooled. The bars were descaled by grinding off any graphite adhesions or surface oxidation products and were cut or ground into physical test specimens. Values for various physical properties are shown in Table B while, the results of oxidation tests are shown in Table C.

Table A

Test No.	Raw Materials	Input Composition Fe-Si-Ti-Al—Others
1	Iron powder + Titanium disilicide	22.53-39.57-36.02-0.87-1.01 (22.96 Fe-40.33 Si-36.71 Ti) ratio.
2	Ferrotitanium + Silicotitanium	24.46-24.10-42.65-3.60-5.19 (26.82 Fe-26.42 Si-46.76 Ti) ratio.
3	Iron powder + Silicotitanium	25.79-33.62-33.83- -6.76 (Al included with "others") (27.66 Fe-36.06 Si-36.28 Ti) ratio.
4	Ferrotitanium + Silicotitanium	23.84-30.65-39.24-0.42-5.85 (25.44 Fe-32.70 Si-41.86 Ti) ratio.
5	Iron, Silicon + Titanium + Titanium disilicide	30.86-33.10-34.85-0.55-0.64 (31.23 Fe-33.50 Si-35.27 Ti) ratio.
6	Iron + Titanium disilicide	22.53-39.57-36.02-0.87-1.01 (22.96 Fe-40.33 Si-36.71 Ti) ratio.
7	Iron + Silicotitanium	25.79-33.62-33.83- -6.76 (Al included with "others") (27.66 Fe-36.06 Si-36.28 Ti) ratio.

Table B.—Physical properties of products

Test No.	Mold Temp. (° C.)	Transverse Rupture (p. s. i., 1" span)		Bulk Density (g./cc.)	Knoop Hardness (100 g. load)
		1,000° C.	1,100° C.		
1	1,250	53,190	----	4.51	1,175
2	1,350	50,059	----	4.80	1,271
3	1,300	----	----	4.73	1,183
4	1,345	----	----	4.76	----
5	1,275	39,868	----	4.63	----
6	1,285	45,520	27,530	4.39	----
7	1,330	29,690	37,220	4.66	----

In tests number 1 to 5 the mold pressure was 4000 p. s. i. and in tests number 6 and 7 the mold pressure was 3000 p. s. i.

The transverse rupture strength was also determined at 1200° C. in tests number 6 and 7 and found to be respectively 16,280 p. s. i. with slight plastic yield at 11,560 p. s. i. and 16,800 p. s. i. with slight plastic yield at 15,090.

Table C.—Oxidation tests, percent growth in air 16 hours at each temperature (cumulative)

Test No.	1,000° C.		1,100° C.		1,200° C.		1,300° C.		1,400° C.		1,500° C.	
	Wt.	Dimension	Wt.	Dimension	Wt.	Dimension	Wt.	Dimension	Wt.	Dimension	Wt.	Dimension
1	.07	.19	.14	.41	.25	1.06	.38	3.41	1.08	5.44	Melted	
2	.57	.96	1.38	1.94	2.81	4.25	14.95	26.23	----	----	----	----
3	.14	.34	.26	.58	.39	.82	.65	1.65	.91	3.76	Melted	
4	.49	.78	.63	1.05	.65	1.25	.84	1.84	1.43	7.52	(1)	(1)
5	.24	.62	.54	1.16	.83	1.63	1.05	2.69	1.40 (Spalling)	4.25	----	----
6	.07	.25	.15	.42	.24	.67	.42	1.78	.79	4.82	----	----
7	.18	.41	.43	.82	.53	1.14	.80	1.73	1.07	7.08	----	----

[1] Strong metal; surface reaction with refractory support.

A sample of the alloy test No. 6 had a tensile strength in air of over 18,500 p. s. i. at 820° C.

A sample of the alloy of test No. 7 withstood 10,280 p. s. i. tensile stress in air at the consecutive temperatures and times as follows before fracture (without measurable elongation):

Temp. Held (Hrs.)	19.63	1.37	21.3	0.35	4.75
Temp. (° C.)	820	820-915	915	915-1,000	1,000

After fabrication and prolonged heat-treatment the alloy objects gave the following analyses:

Test No.	Total Percent Fe+Si+Ti	Ratio Fe-Si-Ti
5	88.93	33.23-32.83-33.94
6	93.78	23.88-40.65-35.46
7	91.41	28.93-36.48-34.59

Although the new products of this invention are composed essentially of iron, silicon and titanium, up to 15%, preferably less than 10%, of other metals and inert diluents that do not materially affect the basic and novel characteristics of the compositions and alloys can be present. Examples are the impurities usually found in minor amount in ferrosilicon, ferrotitanium or silicotitanium alloys such as Al, C, Ca, Mn, and Cr, and metal oxides from attrition of porcelain ball mills. It is preferred that the starting materials contain as small amounts of these impurities, particularly C, as possible.

The final alloys or shaped objects of this invention do not contain appreciable amounts of elemental iron, silicon or titanium although these may constitute a considerable part of the powder metallurgy compositions before firing or hot-pressing. This is very desirable since iron and titanium are generally not stable to high temperature oxidation and elemental silicon is brittle. Some oxidation of the new alloys, particularly the porous alloys, may occur on prolonged heating at high temperature. However, oxidation is not extensive as shown by the small weight gains recorded in the examples and does not materially weaken the structure. In such products complex oxides of iron, silicon and titanium may be present in minor amount. For ease of understanding and simplicity, the compositions listed are those of the initial components, before prolonged heating in air.

In preparing the powder compositions of this invention, elemental iron, silicon, and titanium and/or their binary alloys may be employed. The iron is generally pre-alloyed with silicon while titanium may be pre-alloyed with iron or with silicon. Large amounts of unalloyed elements, particularly silicon and titanium, in the starting materials cause mechanical defects, such as bubbles and cracks and adhesion to mold surfaces during the process of conversion to hard compositions. The elements in controlled proportions, however, are sometimes useful in obtaining alloys which can be fired at the lowest possible initiation temperatures.

Preferably, these powder metallurgy compositions consist of an intimate mixture of silicotitanium or titanium disilicide with iron or with ferrosilicon. Suitable powders are readily obtained by milling or dry-grinding by conventional methods until the desired particle size is achieved. The progress of the grinding or milling may be followed by usual microscopic techniques. Although such powders may contain up to 5% by weight of particles coarser than 75 microns and shaped objects satisfactory for some purposes can be prepared therefrom, it is preferred that less than 2% of such coarse grains be present, i. e., at least 98% by weight of all particles are less than 75 microns. It is still better to screen out all coarse particles, since they serve as points of chemical and mechanical inhomogeneities. Although powders having an average particle size below about 60 microns with at least 25% (by weight) of the particles less than 45 microns in size are preferred, satisfactory shaped alloy objects can be prepared from powders of particle size range 45 to 75 microns and from powders of size range 1 to 45 microns. Iron powder, which is available from commercial sources with a primary particle size of about 1 micron, and commercial ferrosilicons, ferrotitaniums and silicotitaniums which are usually much coarser (e. g., 8–200 mesh) are suitable for use in preparing the compositions of this invention.

The dry powders prepared as above can be shaped by compacting or pressing the powder into the form desired or by slip-casting and drying. Mixtures which contain water or other volatile liquids should be dried before heat-treatment to a liquid content of less than about 3% to prevent rupture of the object during firing.

It is preferred that the shaped objects be no less than about 1 millimeter in minimum dimension since thin objects are not only difficult to obtain but, if obtainable, have low flexural strength and are more subject to oxidation during firing than thicker objects.

The shaped powder compositions are converted into solid alloys by external heating to a temperature of at least 900° C. and preferably 1100–1400° C. The maximum temperature which may be employed is that at which the resultant alloy loses its dimensional stability. In some cases this temperature is as high as 1500° C. The optimum temperature depends upon the exact composition of each powder compact, the properties desired, and the heating method, and may be readily determined by preliminary experiments.

The conversion of the powder composition to solid alloy generally involves calorescence (i. e., an exothermic reaction characterized by an increase in temperature as evidenced by an increase in luminosity) that is induced when a considerable portion of the shaped object is heated to a temperature of at least 900° C. The internal temperature of the shaped object during conversion is generally 1100–1600° C. When the spontaneous heat increase is low, the temperature of the externally applied heat must be higher.

Simple and practical means of effecting this conversion involve heating the shaped powder object with an oxygen-gas torch or placing the object in a hot furnace. When the starting powders are selected from the preferred compositions, the conversion step proceeds readily with minimum weight change, small and predictable dimensional changes, and little deformation of the shaped object. To obtain optimum properties in the resulting alloy, it is preferred that the heating in air be rapid. However, if the green compact contains moisture the initial rate of heating, i. e., up to about 350° C., must be slow to permit drying without cracking. After a temperature of about 350° C. is reached, heating to the initiation temperature of about 900° C. should be as rapid as possible since slow heating in this range brings about powdering, expansion, and cracking of the shaped object before conversion. The time required will depend upon the size of the object being fired; however, for small objects the heating should be accomplished in, at most, 10 minutes and preferably less than 3 to 5 minutes.

As shown in the examples, the alloys of this invention undergo little change in dimensions or weight on heating or firing at temperatures of the order of 1200° C. In contrast, alloys prepared by the same techniques employing compositions near but outside the preferred range with respect to one or more of the elements Fe, Si, or Ti have one or more deficiencies. The following tests (Tables D and E) illustrate this fact.

Table D

Test No.	Ingredients	Input Composition Fe-Si-Ti-Al—Others
1	Steel powder Titanium disilicide	64.28-18.12-15.35-.25 (Al included in "Others") (65.76Fe-18.54Si-15.70Ti) ratio.
2	Iron powder Titanium powder Titanium disilicide	42.38-21.28-36.34 - - (42.38Fe-21.38Si-36.34Ti) ratio.
3	Iron powder Titanium powder Titanium disilicide	42.38-21.28-36.34 - - (42.38Fe-21.38Si-36.34Ti) ratio.
4	Ferrotitanium Titanium disilicide	40.53-23.51-35.19-0.74-0.03 (40.84Fe-23.70Si-35.46Ti) ratio.
5	Titanium Ferrotitanium Titanium disilicide	37.21-12.46-49.66-0.67 - (37.46Fe-12.54Si-50.00Ti) ratio.

Table D—Continued

Test No.	Ingredients	Input Composition Fe-Si-Ti-Al—Others
6	Steel powder, Titanium disilicide	48.18-27.08-23.02- -1.72 (Al included in "Others") (49.02Fe-27.55Si-23.42Ti) ratio.
7	Iron powder, Ferrotitanium, Titanium disilicide	44.36-21.96-30.3-2.46-0.92 (45.91Fe-22.73Si-31.36Ti) ratio.
8	Titanium powder, Ferrotitanium, Silicotitanium	20.49-23.83-47.61-3.00-5.07 (22.29Fe-25.92Si-51.79Ti) ratio.
9	Titanium powder, Silicotitanium, Ferrotitanium	27.46-16.27-48.54-4.08-3.65 (29.76Fe-17.63Si-52.61Ti) ratio.
10	Iron powder, Ferrosilicon, Titanium disilicide	50.07-28.37-19.85-0.48-1.23 (50.94Fe-28.86Si-20.20Ti) ratio.
11	Titanium powder, Ferrotitanium, Titanium disilicide	21.95-13.62-60.0-3.51-0.92 (22.97Fe-14.25Si-62.78Ti) ratio.
12	Iron powder, Titanium disilicide	57.74-21.59-19.65-0.47-0.55 (58.34Fe-21.81Si-19.85Ti) ratio.
13	Iron Powder, Ferrotitanium, Titanium disilicide	41.31—20.05-34.1-3.48-1.06 (43.27Fe-21.00Si-35.73Ti) ratio.
14	Iron Powder, Ferrosilicon, Silicotitanium	39.36-37.91-18.04-0.47-4.22 (41.30Fe-39.77Si-18.93Ti) ratio.

In Table E the abbreviations CP, AF, HP, ΔW, ΔD and T. R. are employed with the following meaning:

CP, AF=cold-pressed, air-fired at temperature (° C.) shown.

HP=hot-pressed at temperature and pressure shown (° C./p. s. i.).

ΔW=weight change and ΔD=dimensional change in the stated temperature range on heating as in Example III.

T. R.=transverse rupture strength (p. s. i.) at temperature (° C.) shown.

Table E

Test No.	Fabrication	Bulk Density (g./cc.)	Knoop Hardness (100 g. Load)	Property Deficiency
1	CP, AF 1,200	4.65		Melts below 1,300°. ΔW=9.83%, 1,000–1,100°.
2	CP, AF 1,100	4.37		Exudes metal below 1,300°. ΔW=20.5%, 1,000–1,200°.
3	HP 1,200/4,000	5.10	1,219	Exudes metal at 1,200°.
4	CP, AF 1,100	4.36	929	Swells at 1,200°. Exudes metal at 1,300°.
5	CP, AF 1,000	3.80		ΔW=24% at 1,000–1,100°. Severe scaling.
6	HP 1,150/4,000	4.47		Weak, T.R. 6,660 at 1,000°.
7	HP 1,205/4,000	5.26	1,437	ΔD=7.82% at 1,000–1,200°. ΔW=11.25% at 1,000–1,300°.
8	HP 1,200/4,000	3.87	1,123	ΔW=9.72% at 1,000–1,200°.
9	HP 1,250/4,000	4.01	974	ΔW=9.77% at 1,000–1,100°.
10	HP 1,300/4,000	5.13		ΔD=17% at 1,000–1,300°.
11	HP 1,180/4,000	4.04		ΔW=25.94% at 1,000–1,200°.
12	HP 1,130/3,000	5.14		Weak. T.R. 10,758 at 1,100°. ΔW=8.91% at 1,000–1,300°.
13	HP 1,200/3,000	4.98		Spalling at 1,100°.
14	HP 1,200/3,000	4.70	1,266	ΔW=7.04%. ΔD=17.28% at 1,000–1,200°.

The new alloys of this invention are hard, resistant to thermal shock and to degradation by heat and oxygen. In fact, heat-treatment of the alloys strengthens and hardens them. Such properties are surprising since some of the alloys have a high percentage of pores of extremely small size. Although upon heating in air at 800–1000° C. a small weight increase takes place, little further increase is noted at temperatures of 1100–1200° C. Thus, these alloy compositions undergo less than a 5% gain or loss (by exudation, for example) in weight and less than 5% change in linear dimensions on heat-treatment in air for 16 hours each at 1000°, 1100° and 1200° C. The preferred alloys show less than 1% weight change and less than 2% dimensional growth on similar heat-treatment. The best alloys show less than 1.5% weight change to 1400° C. and transverse rupture strengths greater than 20,000 p. s. i. at 1000° C. in air. They are extremely resistant to creep under tensile stress in the range of 820–1000° C. Because of these properties, the porous alloys are particularly desirable as starting materials for fabrication of impact-resistant composite structures.

The properties of the new compositions of this invention give them utility in diverse applications. Their thermal stability coupled with electrical resistance in the range of conventional resistance wires and graphite renders them useful in the preparation of electric heating elements. Their hardness, indicated by ability to scratch glass, and ease of fabrication make them useful in the production of tools for cutting and sharpening operations. They also can be fractured into grit size and used in the preparation of bonded abrasive wheels and brazed grit surfaces on metal sheets.

The ease of fabrication of shaped objects is a particular advantage which may favor the use of these alloys over presently used refractories. The conversion to hard products takes place in air thereby avoiding the necessity for vacuum, inert, or reducing atmospheres and the products are easy to make in the form in which they are to be used, e. g., as structural components of high temperature furnaces and heat engines. Furthermore, the precursors of these alloys are commercially available from domestic non-strategic materials.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that this invention is not limited to the specific embodiments thereof except as defined in the appended claims.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

I claim:

1. A powder metallurgy composition consisting of a powder having substantially all particles less than 75 microns in size, and consisting essentially of iron, silicon and titanium in the proportions of 20–45% iron, 25–45% silicon and 25–47.5% titanium.

2. A powder metallurgy composition consisting of a powder having at least 95% by weight of all particles less than 75 microns in size, and consisting essentially of iron, silicon and titanium in the proportions of 20–40% iron, 27.5–42.5% silicon and 30–45% titanium.

3. A powder metallurgy composition consisting of a powder having at least 95% by weight of all particles less than 75 microns in size, and consisting essentially of iron, silicon and titanium in the proportions of 22.5–35% iron, 30–40% silicon and 35–45% titanium.

4. A powder metallurgy composition consisting of a powder having at least 95% by weight of all particles less than 75 microns in size, and consisting essentially of iron, silicon and titanium in the proportions of 22.5–30% iron, 30–37.5% silicon and 35–45% titanium.

5. A powder metallurgy composition consisting of a powder having an average particle size below about 60 microns with at least 25% by weight of the particles being less than 45 microns in size, and consisting essentially of iron, silicon and titanium in the proportions of 20–40% iron, 27.5–42.5% silicon and 30–45% titanium.

6. A powder metallurgy composition of an intimate mixture of ferrosilicon and silicotitanium metal powders having substantially all particles less than 75 microns in size, said intimate mixture of metal powders consisting essentially of iron, silicon and titanium in the proportions of 20–45% iron, 25–45% silicon and 25–47.5% titanium.

7. A powder metallurgy composition of an intimate

mixture of ferrotitanium and silicotitanium metal powders having substantially all particles less than 75 microns in size, said intimate mixture of metal powders consisting essentially of iron, silicon and titanium in the proportions of 20–45% iron, 25–45% silicon and 25–47.5% titanium.

8. A powder metallurgy composition of an intimate mixture of iron and titanium disilicide metal powders having substantially all particles less than 75 microns in size, said intimate mixture of metal powders consisting essentially of iron, silicon and titanium in the proportions of 20–45% iron, 25–45% silicon and 25–47.5% titanium.

9. A shaped object having a minimum dimension of at least 1 mm. composed of an alloy consisting essentially of iron, silicon and titanium in the proportions of 20–45% iron, 25–45% silicon, and 25–47.5% titanium.

10. A shaped object having a minimum dimension of at least 1 mm. composed of an alloy consisting essentially of iron, silicon and titanium in the proportions of 20–40% iron, 27.5–42.5% silicon and 30–45% titanium.

11. A shaped object having a minimum dimension of at least 1 mm. composed of an alloy consisting essentially of iron, silicon and titanium in the proportions of 22.5–35% iron, 30–40% silicon and 35–45% titanium.

12. Process for preparing a strong iron-silicon-titanium object resistant to oxidation and high temperatures which comprises pressing in the form of said object of the powder metallurgy composition of claim 1 and heating said composition to a temperature of at least 900° C.

13. Process for preparing a strong iron-silicon-titanium object resistant to oxidation and high temperatures which comprises pressing in the form of said object the powder metallurgy composition of claim 2 and heating said composition to a temperature of at least 900° C.

14. Process for preparing a strong iron-silicon-titanium object resistant to oxidation and high temperatures which comprises pressing in the form of said object the powder metallurgy composition of claim 3 and heating said composition to a temperature of at least 900° C.

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