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(54) **METHOD OF MAKING AN ALLOY**

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(57) **ABSTRACT**

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A method of making an alloy comprises alloying components comprising: ferrosilicon having a ratio of iron to silicon; and at least one of a metallic element or a metallic compound. The alloy may be used in electrode compositions for lithium ion batteries.

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METHOD OF MAKING AN ALLOY

TECHNICAL FIELD

[0001] The present disclosure broadly relates to methods for making metallic alloys.

BACKGROUND

[0002] Metallic alloys are used in negative electrodes for lithium ion batteries. Negative electrodes containing such metal alloys generally exhibit higher capacities relative to intercalation-type anodes such as graphite. Typically, silicon-containing alloys are made directly from the respective component elements; for example, by a milling process.

[0003] Alloys containing Fe, Si, Sn, and/or C are useful, for example, as active materials for use as a negative electrode material for a lithium ion battery. Small grain sizes of individual phases in such alloys are typically important for good performance as an active electrode material (i.e., reversible lithiation/delithiation). This can typically be achieved by rapid quenching (e.g., melt spinning or sputtering), or milling (e.g., mechanical alloying).

[0004] Current common practice in the industry uses powdered purified elemental metals as raw materials to fabricate active electrode materials using processes that are typically laborious, time-consuming, and/or costly.

SUMMARY

[0005] In one aspect, the present disclosure provides a method of making an alloy, the method comprising alloying components comprising:

[0006] a first ferrosilicon having a first ratio of iron to silicon; and

[0007] at least one of a metallic element or a metallic compound, wherein the alloy is substantially free of crystallites greater than 50 nanometers in size. Alloys prepared according to the present disclosure may be suitable for use as a negative electrode material in a lithium ion battery.

[0008] In some embodiments, the metallic element or metallic compound comprises at least one of carbon, tin, titanium, zinc, iron, or silicon. In some embodiments, the alloy is amorphous. In some embodiments, alloying comprises milling using milling media. In some embodiments, the alloy comprises tin, iron, and silicon. In some embodiments, the alloy comprises tin, iron, and carbon.

[0009] In some embodiments, the metallic compound comprises a second ferrosilicon having a second ratio of iron to silicon, and wherein the first ratio and the second ratio are different.

[0010] Advantageously, alloys prepared according to the present disclosure can typically be prepared easier, faster, and less expensively than with conventional processes that use pure forms of the components used to make the alloy. In part, this is because some grades of ferrosilicon (e.g., iron containing 50, 75, and 90 percent by weight of silicon) are commercially available by the ton at relatively low prices. Many different grades of ferrosilicon are readily available commercially.

[0011] As used herein:

[0012] the term “alloy” refers to a substance having one or more metallic phases, and comprising two or more metallic elements;

[0013] the term “alloying” refers to a process that forms an alloy;

[0014] the term “delithiation” refer to a process for removing lithium from an electrode material;

[0015] the term “metallic” means of, relating to, or having the characteristics of a metal;

[0016] the term “metallic compound” refers to compounds that include at least one metallic element;

[0017] the term “metallic element” refers to all elemental metals (including tin), silicon, and carbon;

[0018] the term “mill” refers to a device for alloying, grinding, pulverizing, or otherwise breaking down a material into small particles (examples include pebble mills, jet mills, ball mills, rod mills and attritor mills);

[0019] the term “milling” refers to a process of placing a material in a mill and operating the mill to perform alloying, or to grind, pulverize, or break down the material into small or smaller particles; and

[0020] the term “negative electrode” refers to an electrode of a lithium ion battery (often called an anode) where electrochemical oxidation and delithiation occurs during a discharging process.

DETAILED DESCRIPTION

[0021] Ferrosilicon is a metallic alloy of iron and silicon commonly prepared by fusing iron and silica in the presence of carbon in an electric furnace. It is of considerable importance in the manufacture of steel and cast iron. Accordingly, ferrosilicon is available commercially in various weight ratios of iron to silicon covering essentially the entire compositional range. Examples include 0.1:99, 1:99, 5:95, 10:90, 20:80, 30:70, 40:60, 50:50, 60:40, 70:30, 80:20, 90:10, 95:5, 99.9:0.1, although other ratios may also be used. Ferrosilicon with the compositions 50:50 (Fe:Si by weight) and 25:75 (Fe:Si by weight) are widely available in tonnage quantities, and at commodity prices that may be less than the constituent purified metallic elements (i.e., iron and silicon).

[0022] According to the present disclosure, at least one ferrosilicon is alloyed with at least one metallic element and/or metallic alloy. The metallic alloy may be any metallic alloy; for example, a ferrosilicon, an alloy of silicon and tin, or an alloy of carbon and iron. In those embodiments of the present disclosure using two compositionally different ferrosilicon alloys, it is possible to achieve any compositional ratio of iron to silicon that falls between the compositional ratios of the two ferrosilicon alloys. For example, using 50:50 Fe:Si and 25:75 Fe:Si ferrosilicon alloys, it is possible to achieve all compositional ranges between 50 and 75 percent silicon by blending appropriate amounts of each. For values lying outside the corresponding range, pure elemental iron or silicon may be added in an amount that achieves a desired stoichiometry.

[0023] Additional metallic elements and metallic alloys may optionally be incorporated in processes according to the present disclosure, may be readily obtained from commercial sources. Examples include carbon, silicon, tin, and transition metals (e.g., Fe, Ti, Y, V, Cu, Zr, Zn, Co, Mn, Mo, and Ni), and alloys thereof, although other metallic elements and metallic alloys may also be used.

[0024] Alloying may be conducted thermally; for example, in an electric arc furnace. Alloying may also be accomplished by mechanical methods such as, for example, milling. The alloying process may use ingot, chunk, powder, or other forms of ferrosilicon and optional metallic components to be included in that alloy.

[0025] Milling techniques are generally useful for mechanically alloying metallic elements and/or metallic alloys; especially as powders. Examples of suitable milling techniques include jet milling, ball milling (e.g., using a planetary mill, vibrational mill, attritor mill, or a cylindrical or conical vessel). Jet mills abrade particles by impinging them against hard target substrates. Ball mills contain milling media that serve to grind material placed in the mill. Examples of suitable milling media include steel, porcelain, and/or ceramic media, which may be in the form of rods, balls, or other shapes. In general, the process conditions will vary with the type of milling technique used, and will be apparent to those of ordinary skill in the milling art.

[0026] In general, milling should be conducted in a controlled oxygen environment; for example, in an inert gas (e.g., nitrogen, helium, and/or argon) environment. In general, the use of preformed metallic alloys significantly reduces the processing time required for forming desired alloys as compared to using pure metallic elements. For example, methods according to the present disclosure are especially useful for mechanically alloying metallic element powders and/or metallic alloy powders.

[0027] Mechanically alloyed compositions prepared according to the present disclosure are useful, for example, for forming electrode compositions (e.g., negative electrode compositions) for use in lithium ion batteries.

[0028] It may be desirable to use milling conditions that result in few if any crystallites of significant size being present in the resultant alloy. For example, the resultant alloy may be formed such that it is substantially free of crystallites greater than 50 nanometers in size (i.e., the maximum dimension of each crystallite). The resultant alloy may contain less than 5 percent by volume, less than one percent by volume, or even less than 0.1 percent by volume of crystallites greater than 50 nanometers in size. In some embodiments, the resultant alloy may be formed as an amorphous composition.

[0029] One desirable method of mechanically alloying metallic elements and/or metallic alloys is described in U.S. application Ser. No. _____, entitled "LOW ENERGY MILLING METHOD, ALLOY, AND NEGATIVE ELECTRODE COMPOSITION" (Attorney Docket No. 65465US002), filed contemporaneously herewith.

[0030] Exemplary alloys include silicon alloys wherein the active material comprises from about 50 to about 85 mole percent silicon, from about 5 to about 25 mole percent iron, from about 0 to about 12 mole percent titanium, and from about 0 to about 12 mole percent carbon. Exemplary alloys of silicon, iron and additional elements may be found in, for example,

[0031] U.S. Pat. Appl. Publ. Nos. 2005/0031957 A1 (Christensen et al), 2007/0020521 A1 (Obrovac et al.), and 2007/0020522 A1 (Obrovac et al.).

[0032] If mechanical alloying is used, a milling aid may optionally be added to the metallic components being alloyed. Examples of milling aids include one or more saturated higher fatty acids (e.g., stearic acid, lauric acid, and palmitic acid) and salts thereof, hydrocarbons such as mineral oil, dodecane, polyethylene powder. In general the amount of any optional milling aid is less than 5 percent, typically less than 1 percent of the millbase.

[0033] Objects and advantages of this disclosure are further illustrated by the following examples, but the particular mate-

rials and amounts thereof recited in these examples, as well as other conditions and details, should not be construed to unduly limit this disclosure.

EXAMPLES

[0034] Unless otherwise noted, all parts, percentages, ratios, etc. in the Examples and the rest of the specification are by weight.

[0035] The following Abbreviations are used in the Examples.

ABBREVIATION	DESCRIPTION
DEC	Diethyl carbonate from Ferro Corp.
EC	Ethylene carbonate from Ferro Corp., Zachary, LA
Fe1	Iron pieces having irregular shape with a size 12 mm or less, 99.97 percent pure, available from Alfa Aesar.
FEC	Fluoroethylene carbonate from Fujian Chuangxin Science and Develops Co., LTD, Fujian, China
FerroSi50	Ferrosilicon containing 47.92 percent of silicon and 51.35 percent of iron available from Globe Metallurgical, Inc. Crushed and sized to less than 500 micrometers.
FerroSi75	Ferrosilicon containing 73.53 percent of silicon and 24.88 percent by weight of iron available from Globe Metallurgical, Inc., Beverly, OH. Crushed and sized to less than 500 micrometers.
Graphite1	A graphite powder available as TIMREX SFG44 from TIMCAL Ltd., Bodio, Switzerland.
Graphite2	A graphite powder with an average particle size of 24.4 micrometers and a 3.2 m ² /g BET surface area available as MAGE from Hitachi Chemical Co. Ltd., Tokyo, Japan
LiOH-20	A 20 percent solution of lithium hydroxide in water, prepared from LiOH—H ₂ O and deionized water.
LiOH—H ₂ O	Lithium hydroxide monohydrate, 98+%, A.C.S. Reagent available from Sigma-Aldrich Co, St. Louis, MO.
PAA-34	A polyacrylic acid solution having a weight average molecular weight of 250,000 g/mole available as a 34 percent solution in water from Sigma-Aldrich Co.
PAA-Li	A 10 percent polyacrylic acid - lithium salt solution in water prepared by titrating PAA-34 with LiOH-20 until fully neutralized and adding deionized water to obtain the desired 10 percent concentration.
Si1	Silicon pieces with a size of 10 cm or less, 98.4 percent pure, available from Alfa Aesar, Ward Hill, MA.
Sn1	325 mesh tin powder, 99.8 percent pure, available from Alfa Aesar.
Ti1	325 mesh titanium powder, 99.5 percent pure, available from Alfa Aesar.

X-Ray Measurement

[0036] X-ray diffraction patterns were collected using a Siemens Model KRISTALLOFLEX 805 D500 diffractometer equipped with a copper target x-ray tube and a diffracted beam monochromator. The X-ray diffraction patterns were collected using scattering angles between 20 and 60 degrees [two-theta] stepped at 0.05 degrees [two-theta]. The crystalline domain size was calculated from the width of x-ray diffraction peaks using the Scherrer equation.

Comparative Example A

[0037] Large grain SiFe alloy was prepared by arc melting 120.277 grams (g) of Si1 and 79.723 g of Fe1. The Si₇₅Fe₂₅ ingot, containing about 75 mole percent silicon and 25 mole percent iron, was crushed and sized to less than 150 micrometers. The Si/Fe large grain alloy powder (120 g), was placed in the 5 liter, steel chamber of a ball mill (Model 611, Jar size 1

available from U.S. Stoneware, Ohio). The chamber was cylindrical in shape with an internal diameter of about 18.8 cm (7.4 inch) and a length of about 17.1 cm (6.75 inch). In addition to the large grain alloy powder, 10 kg of 1.27 cm (0.5 inch) diameter chromium steel balls, one cylindrical steel bar 23.2 cm (9.125 inch) length $\times$ 1.27 cm (0.5 inch) diameter and two cylindrical steel bars 21.5 cm (8.625 inch) length $\times$ 1.27 cm (0.5 inch) diameter were added to the chamber. The chamber was purged with N_2 and milled at 85 rpm (revolutions per minute) for 6 days. After milling, a Si/Fe powder alloy containing about 75 mole percent silicon and 25 mole percent iron was produced (Powder A).

[0038] An X-ray diffraction pattern of the $Si_{75}Fe_{25}$ alloy ingot showed crystalline Si and crystalline $FeSi_2$. An X-ray diffraction pattern of Powder A showed nanostructures with small grain size for the $FeSi_2$ phase and a virtually amorphous Si phase. The term "amorphous" is used in the Examples to describe materials that have no defined X-ray diffraction peak indicative of a crystalline phase.

Example 1

[0039] FerroSi75 (59.27 g) and FerroSi50 (72.15 g) were milled using the procedure described in Comparative Example A, except the milling time was 7 days. After milling, a Si/Fe alloy powder containing about 75 mole percent silicon and 25 mole percent iron was produced, Powder 1. The X-ray diffraction pattern of the starting FerroSi75 showed peaks characteristic of crystalline Si and $FeSi_2$ phases. The X-ray diffraction pattern of the starting FerroSi50 showed peaks characteristic of crystalline $FeSi_2$ phases with small trace of crystalline Si. The x-ray diffraction pattern of Powder 1 showed peaks characteristic of nanocrystalline $FeSi_2$ having a grain size less than 50 nm. The X-ray diffraction pattern of Powder 1 did not contain peaks from Si, indicating that the Si phase in the ball milled alloy was amorphous.

Example 2

[0040] FerroSi75 (83.74 g), 45.55 g of FerroSi50, and 2.38 g of Graphite1 were milled using the procedure described in Comparative Example A, except the milling time was 9 days. After milling, a Si/Fe/C alloy powder containing about 75 mole percent silicon, 20 mole percent iron and 5 mole percent carbon was produced, Powder 2. The X-ray diffraction pattern of Powder 2, showed peaks characteristic of nanocrystalline $FeSi_2$ with a grain size less than 50 nm. The X-ray diffraction pattern of Powder 2 did not contain peaks from Si, indicating that the Si phase in the alloy was amorphous. From stoichiometry, this alloy also contained a SiC phase, however, the X-ray diffraction pattern of the ball milled alloy did not contain peaks from SiC, indicating that this phase was amorphous.

Example 3

[0041] FerroSi75 (111.70 g), 15.08 g of FerroSi50, and 5.10 g of Graphite1 were milled using the procedure described in Example 2. After milling, a Si/Fe/C alloy powder containing about 75 mole percent silicon, 15 mole percent iron and 10 mole percent carbon was produced, Powder 3. The x-ray diffraction pattern of Powder 3 showed peaks characteristic of nanocrystalline $FeSi_2$ with a grain size less than 50 nm. The x-ray diffraction pattern of Powder 3 did not contain peaks from Si, indicating that the Si phase in the alloy was amorphous. From stoichiometry, this alloy also con-

tained a SiC phase, however, the X-ray diffraction pattern of the alloy did not contain peaks from SiC, indicating that this phase was amorphous.

Example 4

[0042] FerroSi75 (46.21 g), 69.06 g of FerroSi50, and 15.97 g of Sn1 were milled using the procedure described in Comparative Example A. After milling, a Si/Fe/Sn alloy powder containing about 71 mole percent silicon, 25 mole percent iron and 4 mole percent tin was produced, Powder 4. The X-ray diffraction pattern of Powder 4 showed peaks characteristic of nanocrystalline $FeSi_2$ with a grain size less than 50 nm. The x-ray diffraction pattern of Powder 4 did not contain peaks from Si and Sn, indicating that the Si and Sn phases in the ball milled alloy were amorphous.

Example 5

[0043] FerroSi75 (46.02 g), 62.82 g of FerroSi50, 16.89 g of Sn1, and 4.27 g of Graphite 1 were placed in the chamber of the ball mill described in Comparative Example A. Milling used the procedure described in Comparative Example A. After milling, a Si/Fe/Sn/C alloy powder containing about 64 mole percent silicon, 22 mole percent iron, 4 mole percent tin and 10 mole percent carbon was produced, Powder 5. The X-ray diffraction pattern of Powder 5 showed peaks characteristic of nanocrystalline $FeSi_2$ with a grain size less than 50 nm. The X-ray diffraction pattern of Powder 5 did not contain peaks from Si, Sn and SiC, indicating that the Si, Sn and SiC phases in the alloy were amorphous.

Example 6

[0044] FerroSi75 (64.29 g), 42.77 g of FerroSi50, 16.14 g of Sn1, and 8.14 g of Ti1 were milled using the procedure described in Comparative Example A, except the milling time was 13 days. After milling, a Si/Fe/Sn/Ti alloy powder containing about 71 mole percent silicon, 20 mole percent iron, 4 mole percent tin and 5 mole percent titanium was produced, Powder 6. The X-ray diffraction pattern of Powder 6, showed peaks characteristic of nanocrystalline $FeSi_2$ with a grain size less than 50 nm. The X-ray diffraction pattern of Powder 6 did not contain peaks from Si, Sn and $TiSi_2$ (and/or $FeTiSi_2$), indicating that the Si, Sn and $TiSi_2$ (and/or $FeTiSi_2$) phase alloy were amorphous.

Example 7

[0045] FerroSi75 (1.27 g), 0.69 g of FerroSi50, and 0.04 g of Graphite1 were placed in a 45-milliliter tungsten carbide chamber, Model 8001 from Spex Certiprep Ltd., Metuchen, N.J. In addition to the powder, 28 tungsten carbide balls (about 108 g) having a 0.79 cm (0.3125 inch) diameter were added to the chamber. The chamber was placed in a dual mixer/mill, Model 8000-D from Spex Certiprep Ltd. The chamber was vibrated by the dual mixer/mill for two hours. The vessel was cooled with an air jet during the two hour milling period, maintaining a chamber temperature of about 30° C. After milling, a Si/Fe/C alloy powder containing about 75 mole percent silicon, 20 mole percent iron, and 5 mole percent carbon was produced, Powder 7. The x-ray diffraction pattern of Powder 7 showed peaks characteristic of nanocrystalline Si and $FeSi_2$ with a grain size 6 nm and 12 nm, respectively. From stoichiometry, this alloy also contained

SiC phases, however, the X-ray diffraction pattern of Powder 7 did not contain peaks from SiC, indicating that this phase was amorphous.

Example 8

[0046] FerroSi75, 1.70 g, and FerroSi50, 0.23 g and 0.08 g Graphite 1 were milled using the procedure described in Example 7. After milling, a Si/Fe/C alloy powder containing about 75 mole percent silicon, 15 mole percent iron and 10 mole percent carbon was produced, Powder 8. The x-ray diffraction pattern of Powder 8 showed peaks characteristic of nanocrystalline Si and FeSi₂ with a grain size of 11 nm and 12 nm, respectively. From stoichiometry, this alloy also contained a SiC phase, however, the X-ray diffraction pattern of Powder 8 did not contain peaks from SiC, indicating that this phase was amorphous.

Procedure for Preparing an Alloy Electrode, Cell Assembly and Cell Testing

[0047] Alloy powder (1.84 g) and 1.6 g of PAA-Li were mixed in a 45-milliliter stainless steel vessel using four, 1.27 cm (0.5 inch) tungsten carbide balls. The mixing was done in a Planetary Micro Mill Pulverisette 7 from Fritsch, Germany at speed 2 for one hour. The resulting solution was hand spread onto a 10-micrometer thick Cu foil using a gap die (typically 3 mil gap). The sample was then dried in a vacuum oven at 120° C. for 1-2 hours producing an alloy electrode film. Circles, 16 mm in diameter, were then punched out of the alloy electrode film and were used as an alloy electrode for a cell (below).

[0048] Half coin cells were prepared using 2325 button cells. All of the components were dried prior to assembly and the cell preparations were done in a dry room with a -70° C. dew point. The cells were constructed from the following components and in the following order, from the bottom up. Cu Spacer/Li metal film/Separator/alloy electrode/Cu spacer. Each cell consisted of a 20 mm diameter×0.762 mm (30 mil) thick disk of Cu spacer, a 16 mm diameter disk of alloy electrode, a 20 mm diameter micro porous separators (CELGAR2400p available from Separation Products, Hoechst Celanese Corp., Charlotte, N.C.), 18 mm diameter×0.38 mm thick disk of Li metal film (lithium ribbon available from Aldrich Chemical Co., Milwaukee, Wis.) and a 20 mm diameter×0.762 mm (30 mil) disk of copper spacer. The electrolyte was a solution containing 90 percent by weight of an EC/DEC solution (2/1 by volume) and 10 percent by weight FEC with LiPF₆ used as the conducting salt at a 1 M concentration. Prior to adding the LiPF₆, the mixture was dried over molecular sieve (3A type) for 12 hours. The cell was filled with 100 microliters of electrolyte solution. The cell was crimp sealed prior to testing.

[0049] Cells were cycled from 0.005V to 0.90V at specific rate of 100 mA/g-alloy with trickle down to 10 mA/g at the end of discharge (delithiation) for the first cycle. From then on, cells were cycled in the same voltage range but at 200 mA/g-alloy and trickle down to 20 mA/g-alloy at the end of discharge. Cells were allowed 15 min rest at open circuit at the end of every half cycle.

Example 9

[0050] An alloy electrode film and three coin cells were prepared and tested according to the *Procedure for Preparing*

an Alloy Electrode, Cell Assembly and Cell Testing using Powder 1. Results are reported Table 1.

Example 10

[0051] An alloy electrode film and three coin cells were prepared and tested according to the *Procedure for Preparing an Alloy Electrode, Cell Assembly and Cell Testing* using Powder 4. Results are reported Table 1.

Example 11

[0052] An alloy electrode film and three coin cells were prepared and tested according to the *Procedure for Preparing an Alloy Electrode, Cell Assembly and Cell Testing* using Powder 5. Results are reported Table 1.

Example 12

[0053] An alloy electrode film and three coin cells were prepared and tested according to the *Procedure for Preparing an Alloy Electrode, Cell Assembly and Cell Testing* using Powder 6. Results are reported Table 1.

Example 13

[0054] An alloy electrode film was prepared using Powder 4. Powder 4 was first sized by milling in heptane. Powder 4 (4 g) and 20 g of heptane were placed in a 45-milliliter stainless steel vessel containing 17.62 g of 9.5 mm (0.375 inch) diameter chromium steel balls and 22.98 g of 6.35 mm (0.25 inch) diameter chromium steel balls. The milling was done in a Planetary Micro Mill Pulverisette 7 at a speed 5 for one hour. Excess heptane was removed, and the wet sample was dried at 80° C. for 1 hour. An alloy electrode film was prepared from the sized powder in the following manner. Sized Powder 4 (0.96 g), 0.96 g of Graphite2, 0.8 g of PAA-Li, and 4.5 g of deionized water, were mixed in a 45-milliliter stainless steel vessel using four 12.7 mm (0.5 inch) diameter tungsten carbide balls. The mixing was done in the Planetary Micro Mill Pulverisette 7 at speed 2 for one hour. The resulting solution was hand spread onto a 10-micrometer thick Cu foil using an 8-mil gap die. The sample was air dried, then calendered and dried in a vacuum oven at 120° C. for 1 hour. Circles, 16 mm in diameter, were then punched out of the alloy electrode film and were used as an alloy electrode for a cell. Three coin cells were prepared according to the second paragraph of the *Procedure for Preparing an Alloy Electrode, Cell Assembly and Cell Testing*. The cells were cycled from 0.005 V to 0.90 V at specific rate of 75 mA/g-alloy with trickle down to 7.5 mA/g at the end of discharge (delithiation) for the first cycle. From then on, cells were cycled in the same voltage range but at 150 mA/g-alloy and trickle down to 15 mA/g-alloy at the end of discharge. Cells were allowed 15 min rest at open circuit at the end of every half cycle. Results are reported in Table 1 (below).

TABLE 1

Example	Initial Capacity Loss, percent	Capacity at Cycle 2, mAh/g	Capacity at Cycle 50, mAh/g	Efficiency percent
9	11	912	895	98
10	13	818	763	93
11	13	861	752	87

TABLE 1-continued

Example	Initial Capacity Loss, percent	Capacity at Cycle 2, mAh/g	Capacity at Cycle 50, mAh/g	Efficiency percent
12	14	875	865	99
13	16	554	544	98

In Table 1 (above), Efficiency = Capacity at Cycle 50/Capacity at Cycle 2.

[0055] Overall, the alloy powders prepared according to the present disclosure, when fabricated into an electrode and further fabricated into a cell, showed stable capacity for many cycles, making them suitable for use as active anode materials in battery applications, including rechargeable lithium ion battery applications.

[0056] All patents and publications referred to herein are hereby incorporated by reference in their entirety. All examples given herein are to be considered non-limiting unless otherwise indicated. Various modifications and alterations of this disclosure may be made by those skilled in the art without departing from the scope and spirit of this disclosure, and it should be understood that this disclosure is not to be unduly limited to the illustrative embodiments set forth herein.

What is claimed is:

1. A method of making an alloy, the method comprising alloying components comprising:

a first ferrosilicon having a first ratio of iron to silicon; and at least one of a metallic element or a metallic compound, wherein the alloy is substantially free of crystallites greater than 50 nanometers in size.

2. The method of claim 1, wherein said metallic element or metallic compound comprises at least one of carbon, tin, titanium, zinc, iron, or silicon.

3. The method of claim 1, wherein the alloy is amorphous.

4. The method of claim 1, wherein alloying comprises milling using milling media.

5. The method of claim 1, wherein the alloy comprises tin, iron, and silicon.

6. The method of claim 1, wherein the alloy comprises tin, iron, and carbon.

7. The method of claim 1, wherein the metallic compound comprises a second ferrosilicon having a second ratio of iron to silicon, and wherein the first ratio and the second ratio are different.

8. The method of claim 7, wherein the components further comprise tin.

9. The method of claim 7, wherein the components further comprise titanium.

10. The method of claim 1, wherein the alloy is adapted for use as an active material in a negative electrode composition in a lithium ion battery.

11. The method of claim 10, wherein said metallic element or metallic compound comprises at least one of carbon, tin, titanium, zinc, iron, or silicon.

12. The method of claim 10, wherein the alloy is amorphous.

13. The method of claim 10, wherein alloying comprises milling using milling media.

14. The method of claim 10, wherein the alloy comprises tin, iron, and silicon.

15. The method of claim 10, wherein the alloy comprises tin, iron, and carbon.

16. The method of claim 10, wherein the components further comprise a second ferrosilicon having a second ratio of iron to silicon, and wherein the first ratio and the second ratio are different.

17. The method of claim 16, wherein the components further comprise tin.

18. The method of claim 16, wherein the components further comprise titanium.

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