(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
5 October 2017 (05.10.2017)

(10) International Publication Number
WO 2017/172748 A1

(51) International Patent Classification:
C01B 33/021 (2006.01) *B65G 53/04* (2006.01)

(21) International Application Number:
PCT/US2017/024512

(22) International Filing Date:
28 March 2017 (28.03.2017)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
62/315,334 30 March 2016 (30.03.2016) US

(71) Applicant: **SITEC GMBH** [DE/DE]; Buergermeister-Wegele-Strasse 12, 86167 Augsburg (DE).

(72) Inventors: **DASSEL, Mark, W.**; 20699 Kitsap Street Northeast, Indianola, Washington 98342 (US). **KERAT, Uwe**; c/o SiTec GmbH, Buergermeister-Wegele-Strasse 12, 86167 Augsburg (DE).

(74) Agents: **ABRAMONTE, Frank** et al.; Seed Intellectual Property Law Group LLP, Suite 5400, 701 Fifth Avenue, Seattle, Washington 98104-7064 (US).

(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DJ, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Declarations under Rule 4.17:
— *of inventorship (Rule 4.17(iv))*

Published:
— *with international search report (Art. 21(3))*

(54) Title: SYSTEMS AND METHODS FOR DUST SUPPRESSED SILICON CHARGING IN A VACUUM

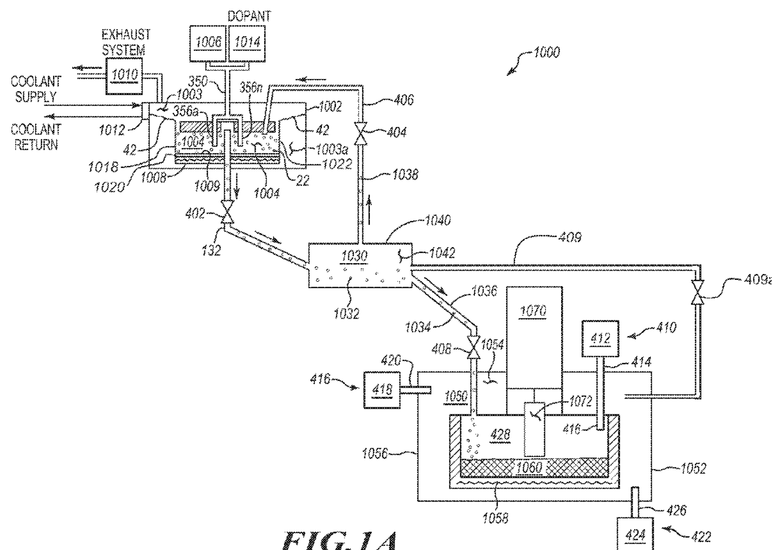

FIG.1A

(57) Abstract: A crystal production system and method that maintains a high vacuum during both a melting phase and a charging phase enables improved dust suppression. In such a system and method, a particulate feed vessel and a crucible inside a melting chamber are fluidly connected. Silicon granules are conveyed from the particulate feed vessel to the crucible inside the melting chamber. The silicon granules are then melted in the crucible within the melting chamber. The dust particles are suppressed by charging the melted silicon under the high vacuum. Charging can occur while performing a gaseous purge, with limiting a gaseous purge, or while forgoing any gaseous purge in the melting chamber. Doping can occur, for example in the charging chamber or crucible by addition of dopant in either particulate or gaseous forms.

WO 2017/172748 A1

SYSTEMS AND METHODS FOR DUST SUPPRESSED SILICON CHARGING IN A VACUUM

TECHNICAL FIELD

This disclosure generally relates to mechanically vibrated reactors
5 and associated crystal production methods.

BACKGROUND

Silicon, specifically polysilicon, is a basic material from which a large variety of semiconductor products are made. Silicon forms the foundation of many integrated circuit technologies, as well as photovoltaic transducers. Of
10 particular industry interest is high purity silicon.

Processes for producing polysilicon may be carried out in different types of reaction devices, including chemical vapor deposition reactors and fluidized bed reactors. Various aspects of the chemical vapor deposition (CVD) process, in particular the Siemens or “hot wire” process, have been described,
15 for example in a variety of U.S. patents or published applications (*see,* e.g., U.S. Patent Nos. 3,011,877; 3,099,534; 3,147,141; 4,150,168; 4,179,530; 4,311,545; and 5,118,485).

Silane and trichlorosilane are both used as feed materials for the production of polysilicon. Silane is more readily available as a high purity
20 feedstock because it is easier to purify than trichlorosilane. Production of trichlorosilane introduces boron and phosphorus impurities, which are difficult to remove because they tend to have boiling points that are close to the boiling point of trichlorosilane itself. Although both silane and trichlorosilane are used as feedstock in Siemens-type chemical vapor deposition reactors,
25 trichlorosilane is more commonly used in such reactors. Silane, on the other hand, is a more commonly used feedstock for production of polysilicon in fluidized bed reactors.

Silane has drawbacks when used as a feedstock for either chemical vapor deposition or fluidized bed reactors. Producing polysilicon from silane in a Siemens-type chemical vapor deposition reactor may require up to twice the electrical energy compared to producing polysilicon from
5 trichlorosilane in such a reactor. Further, the capital costs are high because a Siemens-type chemical vapor deposition reactor yields only about half as much polysilicon from silane as from trichlorosilane. Thus, any advantages resulting from higher purity of silane are offset by higher capital and operating costs in producing polysilicon from silane in a Siemens-type chemical vapor deposition
10 reactor. This has led to the common use of trichlorosilane as feed material for production of polysilicon in such reactors.

Silane as feedstock for production of polysilicon in a fluidized bed reactor has advantages regarding electrical energy usage compared to production in Siemens-type chemical vapor deposition reactors. However,
15 there are disadvantages that offset the operating cost advantages. In using the fluidized bed reactor, the process itself may result in a lower quality polysilicon product even though the purity of the feedstock is high. For example, polysilicon produced in a fluidized bed reactor may also include metal impurities from the equipment used in providing the fluidized bed due to the typically
20 abrasive conditions found within a fluidized bed. Further, polysilicon dust may be formed, which may interfere with operation by forming ultra-fine particulate material within the reactor and may also decrease the overall yield. Further, polysilicon produced in a fluidized bed reactor may contain residual hydrogen gas, which must be removed by subsequent processing. Thus, although high
25 purity silane may be available, the use of high purity silane as a feedstock for the production of polysilicon in either type of reactor may be limited by the disadvantages noted.

Chemical vapor deposition reactors may be used to convert a first chemical species, present in vapor or gaseous form, to solid material. The
30 deposition may and commonly does involve the conversion or decomposition of

the first chemical species to one or more second chemical species, one of which second chemical species is a substantially non-volatile species.

Decomposition and deposition of the second chemical species on a substrate is induced by heating the substrate to a temperature at which the
5 first chemical species decomposes on contact with the substrate to provide one or more of the aforementioned second chemical species, one of which second chemical species is a substantially non-volatile species. Solids so formed and deposited may be in the form of successive annular layers deposited on bulk forms, such as immobile rods, or deposited on mobile substrates, such as
10 beads, grains, or other similar particulate matter chemically and structurally suitable for use as a substrate.

Beads or particles are currently produced, or grown, in a fluidized bed reactor where an accumulation of dust, comprised of the desired product of the decomposition reaction, acting as seeds for additional growth, and pre-
15 formed beads, also comprised of the desired product of the decomposition reaction, are suspended in a gas stream passing through the fluidized bed reactor. Due to the high gas volumes needed to fluidize the bed within a fluidized bed reactor, where the volume of the gas containing the first chemical species is insufficient to fluidize the bed within the reactor, a supplemental
20 fluidizing gas such as an inert or marginally reactive gas is used to provide the gas volume necessary to fluidize the bed. As an inert or only marginally reactive gas, the ratio of the gas containing the first chemical species to the supplemental fluidizing gas may be used to control or otherwise limit the reaction rate within or the product matrix provided by the fluidized bed reactor.
25 The use of a supplemental fluidizing gas however can increase the size of process equipment and also increases separation and treatment costs to separate any unreacted or decomposed first chemical species present in the gas exiting the fluidized bed reactor from the supplemental gas used within the fluidized bed reactor.

In a conventional hydraulically fluidized bed reactor, silane and one or more diluents such as hydrogen are used to fluidize the bed. Since the fluidized bed temperature is maintained at a level sufficient to thermally decompose silane, the gases used to fluidize the bed, due to intimate contact with the bed, are necessarily heated to the same bed temperature. For example, silane gas fed to a fluidized bed reactor operating at a temperature exceeding 500ºC is itself heated to its auto-decomposition temperature. This heating causes some of the silane gas to undergo spontaneous thermal decomposition which creates an extremely fine (e.g., having a particle diameter of 5 microns or less, or 1 micron or less, or 0.1 micron or less) silicon powder that is often referred to as “amorphous dust” or “poly-powder.” Silane forming poly-powder instead of the preferred polysilicon deposition on a substrate represents lost yield and unfavorably impacts production economics. The very fine poly-powder is electrostatic and is fairly difficult to separate from product particles for removal from the system. Additionally, if the poly-powder is not separated, off-specification polysilicon granules (i.e., polysilicon granules having a particle size less than the desired diameter of about 1.5mm) are formed, further eroding yield and further unfavorably impacting production economics.

In some instances, a silane yield loss to poly-powder is on the order of about 15%, but may range from about 10% to about 20%. The poly-powder particle size is typically about 0.1 micron, but can range from about 0.05 microns to about 10 microns. A 1% yield loss can therefore create around 1×10^{16} poly-powder particles. Unless these fine poly-powder particles are removed from the fluidized bed, the poly-powder will provide particles having only 1/3,000th of the industry desired diameter of 1.5 mm. Thus the ability to efficiently remove ultra-fine particles from the fluidized bed or from the fluid bed reactor off-gas is important. However, electrostatic forces often hinder filtering the ultra-fine poly-powder from a finished product or fluid bed reactor off-gas.

Therefore, processes that minimize or ideally avoid the formation of the ultra-fine poly-powder are quite advantageous.

The silicon coated particles produced in the reactor are typically removed, and packaged for commercial shipment to producers of silicon boules that are used to manufacture a wide variety of semiconductor products. The handling, storage, and shipment of such silicon coated particles expose the particles to atmospheric oxygen which quickly forms an oxide layer or shell on the exposed surfaces of the particles. This oxide layer adversely impacts the melting process and introduces unacceptable levels of contaminants into the crystal production process. As such, this oxide layer negatively impacts productivity and quality.

Problems due to dusting in the melting process

Dusting is a problem that may occur when silicon granules are poured into a crucible that is already in place inside the melting chamber. This issue may occur during the initial charge or the recharge with granules.

The exhaust gases exiting Czochralski pullers, which are used for growing monocrystalline silicon ingots, contain dust particles. This dust needs to be filtered out to protect the surrounding people, the environment, and the system vacuum pumps. Some of the dust is present in the feed granules, while other of the dust is formed by condensation of by-product materials made in the melting process above the silicon melt (e.g., SiO released from the melt can combine to produce micronic or sub-micronic solid particles). Fine silicon dust is highly reactive and immediately oxidizes to SiO and SiO2 in an exothermic reaction. Accordingly, this dust can be very hazardous.

Granulated polysilicon has a porous structure resulting in two disadvantageous properties: (1) gas is included in the pores; and (2) granulated polysilicon is prone to abrasion. Since gas is included in the pores, this gas is released during melting and disrupts the further processing of the granulated polysilicon. It is thus, useful to reduce the gas content of the granulated polysilicon. However, this requires an additional working step, which increases

production costs and, moreover, results in additional contamination of the granulated polysilicon.

Continuing, since granulated polysilicon is prone to abrasion, fine silicon dust is formed during handling of the granulated polysilicon, e.g., during production of the granulated polysilicon in the fluidized bed, as well as when the granulated polysilicon is being transported to the user. This dust is disruptive in a number of ways. For example, the dust interferes with transportation of the granulated polysilicon within the production installation because it causes deposits to form on pipelines and leads to blockages in valves and fittings. Additionally, the dust interferes with the further processing of the granulated polysilicon when the dust floats on the surface of the molten polysilicon while the granulated polysilicon is being melted. In this manner, gaseous drafts within the CZ (*Czochralski*) melter/puller apparatus can carry dust onto the surface of the monocrystal, thereby causing defects. Further, dust is a potential contamination carrier due to its large specific surface area.

Granular production is typically based on monosilane as the silicon-containing starting gas. In addition to dust caused by the abrasion in the deposition process and subsequent handling steps, the fluidized bed reactor process may also result in the direct formation of dust due to a homogeneous gas phase reaction. This dust may be blown out of the fluidized bed reactor into overhead filters or stay with the granules due to electrostatic or other attractive forces.

The dust accumulates in the CZ melter/puller apparatus and may be transported by an argon gas flow (or other inert gas flow) toward the growing crystal. Accumulated dust can at times break off of internal surfaces, falling on to the surface of the molten pool from which crystal is drawn. This dust issue can cause defect generation or structure loss, with larger dust particles leading to more serious defects. This is a significant problem, especially during the crucible feeding process. Additionally, the dust is harmful to valves, seals, O-rings, and other components. The dust particles continue to be a problem during the monocrystal production process since the dust particles are difficult

to melt, especially if they are covered by an oxide layer. Additionally, since the surface to volume ratio of dust particles is high, dust particles easily accumulate impurities, resulting in an overall higher impurity level. Finally, the operators do not like dusty, dirty material, and low acceptance by the operators results in less careful operation of the furnace. Many researchers of international stature have commented on the problem of dusting without proposing an adequate solution.

BRIEF SUMMARY

A dust suppression silicon production system and method may be summarized as including a particulate feed vessel and a crucible inside a melting chamber. Such maintains a high vacuum (e.g., 10 millibar to 100 millibar) during both a melting phase and a charging phase and enables dust suppression due to the lack of “lift” in a high vacuum. The particulate feed vessel and the crucible inside a melting chamber may advantageously be fluidly connected, for example via a conduit. Silicon granules are conveyed from the particulate feed vessel to the crucible inside the melting chamber via the conduit. The silicon granules are then melted in the crucible within the melting chamber. The dust particles are suppressed by charging the melted silicon under the high vacuum. Such may, for example, occur while either i) normal gaseous purging, ii) restricted gaseous purging, or iii) forgoing any gaseous purging during the charging phase to maintain the high vacuum. The silicon granules added to the melter may be pre-doped (i.e., doped prior to being supplied to the charging chamber, melter, or crucible). Alternatively, a doping subsystem may, for example, be fluidly coupled and operable to supply a dopant to the charging chamber or interior of the melter and/or the crucible in a gaseous or particulate (e.g., powder, chunks) form.

The high vacuum can, for example, be 300 Torr or less. For instance, the high vacuum can be 200 Torr or less. For instance, the high vacuum can be 100 Torr or less. For instance, the high vacuum can be 20 Torr or less. For instance, the high vacuum can be 10 Torr or less.

The dust particles can, for example, be 500 microns or less in diameter. For instance, the dust particles can be 300 microns or less in diameter. For instance, the dust particles can be 100 microns or less in diameter. For instance, the dust particles can be 20 microns or less in
5 diameter. For instance, the dust particles can be 10 microns or less in diameter. For instance, the dust particles can be 1 micron or less in diameter.

A dust suppression inducing crystal production method may be summarized as including: maintaining a high vacuum within a particulate feed vessel and a melting chamber; fluidly connecting the particulate feed vessel
10 and a crucible inside the melting chamber; conveying silicon granules from the particulate feed vessel to the crucible inside the melting chamber; melting the silicon granules in the crucible inside the melting chamber; and enabling suppression of dust particles by charging the melted silicon under the maintained high vacuum. Doping may occur, for example by supplying a
15 dopant to the charging chamber or interior of the melter and/or the crucible in a gaseous or particulate form.

Dustless or reduced dust transfer advantageously helps make possible the use of smaller diameter particles in a melter than might otherwise be employed. Preventing the formation of SiO_2 may also advantageously
20 facilitate the use of smaller diameter particles in a melter than might otherwise be employed.

BRIEF DESCRIPTION OF SEVERAL VIEWS OF THE DRAWINGS

In the drawings, identical reference numbers identify similar elements or acts. The sizes and relative positions of elements in the drawings
25 are not necessarily drawn to scale. For example, the shapes of various elements and angles are not drawn to scale, and some of these elements are arbitrarily enlarged and positioned to improve drawing legibility. Further, the particular shapes of the elements, as drawn, are not intended to convey any

information regarding the actual shape of the particular elements, and have been solely selected for ease of recognition in the drawings.

Figure 1A is a schematic view of an illustrative crystal production system in which a reactor containing a particulate bed produces coated particles that are transported via a conduit to a coated particle melter while maintained in a vacuum environment which may be a free oxygen reduced environment, according to an illustrated implementation.

Figure 1B is a block diagram of a configuration in which the conduit includes only a hermetic coupling between a reactor containing a particulate bed from which coated particles are separated and a coated particle melter (i.e., a “close-coupled” configuration), according to an illustrated implementation.

Figure 1C is a block diagram of a configuration in which the conduit includes a coated particle accumulator positioned between a reactor containing a particulate bed from which coated particles are separated and a coated particle melter, according to an illustrated implementation.

Figure 1D is a block diagram of a configuration in which the conduit includes a coated particle classifier positioned between a reactor containing a particulate bed from which coated particles are separated and a coated particle melter, according to an illustrated implementation.

Figure 1E is a block diagram of a configuration in which the conduit includes a coated particle accumulator and a coated particle classifier positioned between a reactor containing a particulate bed from which coated particles are separated and a coated particle melter, according to an illustrated implementation.

Figure 1F is a block diagram of a configuration in which the conduit includes a coated particle classifier and a coated particle grinder positioned between a reactor containing a particulate bed from which coated particles are separated and a coated particle melter, according to an illustrated implementation.

Figure 1G is a block diagram of a configuration in which the conduit includes a coated particle accumulator, a coated particle classifier, and a coated particle grinder positioned between a reactor containing a particulate bed from which coated particles are separated and a coated particle melter, according to an illustrated implementation.

Figure 2 is a schematic view of an illustrative crystal production method in which a reactor, for instance a mechanically vibrated fluidized or packed bed reactor is close coupled and hermetically sealed to a melter that receives coated particles removed from the particulate bed; according to an illustrated implementation.

Figure 3 is a high level flow diagram of an illustrative crystal production method in which coated particles separated from a particulate bed disposed in a reactor are transferred to a coated particle melter in a vacuum environment which may contain a reduced level of free oxygen, according to an illustrated implementation.

Figure 4 is a high level flow diagram of an illustrative crystal production method in which coated particles separated from a fluidized particulate bed disposed in a reactor are transferred to a coated particle melter in a vacuum environment which may contain a reduced level of free oxygen, according to an illustrated implementation.

Figure 5 is a high level flow diagram of an illustrative crystal production method in which coated particles are produced by supplying a thermally decomposable first gaseous chemical species and one or more diluents to a particulate bed, according to an illustrated implementation.

Figure 6 is a high level flow diagram of an illustrative crystal production method in which doped coated particles are produced by supplying a thermally decomposable first gaseous chemical species and one or more dopants to a particulate bed, according to an illustrated implementation.

Figure 7 is a high level flow diagram of an illustrative crystal production method in which a chamber in a reactor containing the particulate

bed is maintained at a temperature below the thermal decomposition temperature of a first gaseous chemical species to limit the decomposition of the first gaseous chemical species external to the particulate bed, according to an illustrated implementation.

5 Figure 8 is a high level flow diagram of an illustrative crystal production method in which coated particles separated from a particulate bed are divided into a first portion and a second portion and at least some of the second portion of coated particles is returned to the particulate bed, according to an illustrated implementation.

10 Figure 9 is a high level flow diagram of an illustrative crystal production method in which coated particles are melted in a coated particle melter from which a crystal puller draws a second chemical species crystal, according to an illustrated implementation.

Figure 10 is a high level flow diagram of an illustrative crystal 15 production method in which coated particles separated from a particulate bed disposed in a reactor are transferred to a coated particle melter in a vacuum environment which may contain a reduced level of free oxygen, according to an illustrated implementation.

Figure 11 is a high level flow diagram of an illustrative crystal 20 production method in which coated particles separated from a particulate bed disposed in a reactor are transferred to a coated particle melter in a vacuum environment which may contain a reduced level of free oxygen, according to an illustrated implementation.

DETAILED DESCRIPTION

25 In the following description, certain specific details are included to provide a thorough understanding of various disclosed embodiments. One skilled in the relevant art, however, will recognize that embodiments may be practiced without one or more of these specific details, or with other methods, components, materials, etc. In other instances, well-known structures

associated with systems for making silicon including, but not limited to, vessel design and construction details, metallurgical properties, piping, control system design, mixer design, separators, vaporizers, valves, controllers, or final control elements, have not been shown or described in detail to avoid unnecessarily obscuring descriptions of the embodiments.

Unless the context requires otherwise, throughout the specification and claims which follow, the word “comprise” and variations thereof, such as, “comprises” and “comprising” are to be construed in an open, inclusive sense, that is, as “including, but not limited to.”

Reference throughout this specification to “one embodiment,” or “an embodiment,” or “another embodiment,” or “some embodiments,” or “certain embodiments” means that a particular referent feature, structure, or characteristic described in connection with the embodiment is included in at least one embodiment. Thus, the appearance of the phrases “in one embodiment,” or “in an embodiment,” or “in another embodiment,” or “in some embodiments,” or “in certain embodiments” in various places throughout this specification are not necessarily all referring to the same embodiment. Furthermore, the particular features, structures, or characteristics may be combined in any suitable manner in one or more embodiments.

It should be noted that, as used in this specification and the appended claims, the singular forms "a," "an," and "the" include plural referents unless the content clearly dictates otherwise. Thus, for example, reference to a chlorosilane includes a single species of chlorosilane, but may also include multiple species of chlorosilanes. It should also be noted that the term "or" is generally employed as including "and/or" unless the content clearly dictates otherwise.

As used herein, the term “silane” refers to SiH_4. As used herein, the term “silanes” is used generically to refer to silane and/or any derivatives thereof. As used herein, the term “chlorosilane” refers to a silane derivative wherein one or more of hydrogen has been substituted by chlorine. The term

"chlorosilanes" refers to one or more species of chlorosilane. Chlorosilanes are exemplified by monochlorosilane (SiH_3Cl or MCS); dichlorosilane (SiH_2Cl_2 or DCS); trichlorosilane ($SiHCl_3$ or TCS); or tetrachlorosilane, also referred to as silicon tetrachloride ($SiCl_4$ or STC). The melting point and boiling point of
5 silanes increases with the number of chlorines in the molecule. Thus, for example, silane is a gas at standard temperature and pressure (0ºC/273 K and 101 kPa), while silicon tetrachloride is a liquid. As used herein, the term "silicon" refers to atomic silicon, *i.e.*, silicon having the formula Si. Unless otherwise specified, the terms "silicon" and "polysilicon" are used
10 interchangeably herein when referring to the silicon product of the methods and systems disclosed herein. Unless otherwise specified, concentrations expressed herein as percentages should be understood to mean that the concentrations are in mole percent.

As used herein, the terms "chemical decomposition," "chemically
15 decomposed," "thermal decomposition," and "thermally decomposed" all refer to a process by which a first gaseous chemical species (e.g., silane) is heated to a temperature above a thermal decomposition temperature at which the first gaseous chemical species decomposes to at least a non-volatile second chemical species (e.g., silicon). In some implementations, the first gaseous
20 chemical species may also yield one or more third gaseous chemical decomposition byproducts (e.g., hydrogen). Such reactions may be considered as a thermally initiated chemical decomposition or, more simply, as a "thermal decomposition." It should be noted that the thermal decomposition temperature of the first gaseous chemical species is not necessarily a fixed value and may
25 vary with the pressure at which the first gaseous chemical species is maintained.

As used herein, the term "mechanically fluidized" refers to the mechanical suspension or fluidization of particles forming the particulate bed, for example by mechanically oscillating or vibrating the particulate bed in a
30 manner promoting the flow and circulation (i.e., the "mechanical fluidization") of

the particles. Such mechanical fluidization, generated by a cyclical physical displacement (e.g., vibration or oscillation) of the one or more surfaces supporting the particulate bed or the retainment volume about the particulate bed, is therefore distinct from liquid or gaseous (i.e., hydraulic) bed fluidization
5 generated by the passage of a liquid or gas through a particulate bed. It should be noted with particularity that a mechanically fluidized particulate bed is not reliant upon the passage of a fluid (i.e., liquid or gas) through the plurality of particulates to attain fluid-like behavior. As such, fluid volumes passed through a mechanically fluidized bed can be significantly smaller than the fluid volumes
10 used in a hydraulically fluidized bed. In addition, a quiescent (i.e., non-fluidized) plurality of particles represents a "settled bed" which occupies a "settled volume." When fluidized, the same plurality of particles occupies a "fluidized volume" which is greater than the settled volume occupied by the plurality of particles. Unless specified otherwise, the terms "vibration" and
15 "oscillation," and variations of such (e.g., vibrating, oscillating) are used interchangeably herein.

As used herein, the terms "mechanically vibrated packed bed" or "mechanically vibrated compressed bed" and variants thereof (e.g., vibrating packed bed) refers to a bed of particles that is mechanical vibrated (e.g.,
20 movement in at least a vertical direction), for example by mechanically oscillating or vibrating the particulate bed in a manner that the preponderance of the particles are in both immediate and sustained contact with each other such that the particles comprising the packed or compressed bed vibrate in three dimensions and rotationally with respect to each other. In contrast, in a
25 static bed the particles are fixed in place and do not vibrate or move with respect to one another. In contrast to a mechanically fluidized bed in which the bed may expand 10% or more, expansion of the vibrated packed or compressed bed is limited or restrained, for instance limited or restrained to 1% to 5% of expansion relative to a static bed. Further, in a mechanically vibrated
30 packed or compressed bed the particles vibrate with respect to each other, and

move relatively slowly, for example at a rate of around 0.5 inches to 1 inch per 60 seconds. In contrast, in a mechanically vibrated fluidized bed the particles move a rate of, for example, around 1 inch to 3 inches per second.

As used herein, the terms “particulate bed” and “heated particulate bed” refer to any type of particulate bed, including vibrated packed or compressed particulate beds, hydraulically fluidized particulate beds, and mechanically fluidized particulate beds. The term “heated fluidized particulate bed” can refer to either or both a heated hydraulically fluidized particulate bed, a heated mechanically fluidized particulate bed, and/or a heated vibrated packed or compressed particulate bed. The term “hydraulically fluidized particulate bed” refers specifically to a fluidized bed created by the passage of a fluid (i.e., liquid or gas) through a particulate bed. The term “mechanically fluidized particulate bed” refers specifically to a fluidized bed created by oscillating or vibrating a surface supporting the particulate bed at an oscillatory frequency and/or oscillatory displacement sufficient to fluidize the particulate bed. The term “mechanically vibrated packed or compressed particulate bed” refers specifically to a bed created by oscillating or vibrating a surface supporting the particulate bed while restraining expansion of the particulate bed, for instance, between 1% to 5% of the height of a static bed of the particles.

Typically, in a fluidized bed reactor (FBR) solid particles are held in a suspended condition by upflowing gases, such that the solids move in a manner typical of liquids. Specifically, the downward weight of the solid mass is exactly balanced by the upward drag force of the gas flow. The fluidization of the solids is achieved by the hydraulics of upflowing gases in a conventional FBR. This is in contrast to some of the more unique reactors described below (Vibrated Fluidized Bed Reactor (VFBR) and Vibrated Packed Bed Reactor (VPBR)). Usually a conventional FBR is hydraulic in nature.

In a fluidized bed reactor, the pressure drop per unit vertical length is almost invariant with changes in gas flow until the gas velocity reaches

the terminal velocity of the particle. Kinetic reactivity is increased greatly in a fluidized bed reactor over that of a static packed bed for solid-gas chemical reactions. Additionally, heat transfer is increased greatly in a fluidized bed reactor over that of gases moving through a static packed bed. Continuing, the Minimum Fluidization Velocity (U_{mf}) is the gas superficial velocity at which the packed bed pressure drop curve (per unit of vertical height) intersects the invariant value of bed pressure drop in the buoyant condition. This characterization of fluidization is only relative to a conventional (hydraulic) fluidized bed reactor.

For particles of a wide mixture of diameters, the pressure drop of the hydraulic fluidized bed reactor's bed may be characterized by the Sauter Mean Diameter = Aerodynamic Average Diameter (i.e., the particle diameter where the surface area/volume ratio is equal to the entire bed's surface area divided by the entire bed's particle volume). There is no analog to the Sauter Mean Diameter in any other type of solid bed than a conventional (hydraulic) fluidized bed reactor, where the upflowing gas exerts a significant drag force on the particles.

There is a relative fluidization degree, measured in multiples of U_{mf}, where the bed remains mixed. Below this relative fluidization, smaller particles are conveyed upward and the larger ones sink to the bottom of the bed. This degree of relative fluidization is co-incident with the onset of bubble formation. Such a bed is considered segregated. Notably, the bed mixing and bed segregation phenomena are different with non-hydraulic types of fluidization.

In a conventional (hydraulic) segregated fluidized bed reactor, the larger particles settle to the bottom but still retain some degree of random motion (i.e., that portion of the bed is not static). The smallest particles in such segregated fluidized bed reactor may reach terminal velocity at the very top of the bed and be ejected out of the bed. Again, this segregation characteristic

applies only to a conventional (hydraulic) fluidized bed reactor. In a non-segregated fluidized bed reactor, the rising gas bubbles coalesce in the center, inducing a toroidal pattern of solids flow (i.e., upward in the center, and downward along the vessel walls). Expanded heads or internal cyclones are often used to control the amount of fine particles ejected from the top of the bed. Special distributor plates and bubble breakers are used to control bubble formation, which is usually considered negatively.

In some implementations, ;hydraulically fluidized bed reactors are used for silane epitaxial reaction to polysilicon granules. In silicon hydraulically fluidized bed reactors, the characteristic bed expansion is about 10%, as the bed transitions from a static bed to a bed just above the Minimum Fluidization Velocity (U_{mf} =1). In a typical mixed size bed of silicon particles, where the relative fluidization is high enough to avoid segregation, the characteristic hydraulically fluidized bed expansion is about 15%. The boundary of segregation in a mixed size bed of silicon particles is between 2-3 times U_{mf}. Notably, the bed expansion is far less in vibrated packed or compressed bed reactors (e.g., about 1% to about 5%) as compared to the 10% or more bed expansion characteristic of vibrated fluidized bed reactors when operated at similar conditions.

In conventional (hydraulic) silicon decomposer fluidized bed reactors in which seed particles of polysilicon are grown by epitaxial deposition, the preferred degree of fluidization is slightly greater than U_{mf} =1, and below U_{mf} =2. This segregates the bed and allows the larger product particles to be withdrawn from the bottom of the reactor in a separate “boot.” In mechanically vibrated fluidized bed reactors and mechanically vibrated packed bed reactors, the product removal is withdrawn differently, and from the top of the reactor.

Since conventional (hydraulic) silicon decomposer fluidized bed reactors require heat input (e.g., generally to hold a 600-700°C range), the relative fluidization must be high enough to allow reasonable heat flux at the

vessel walls. Impurity concerns prevent the use of internal structures to provide heat input. Also, the relative fluidization must be high enough to maintain an adequate degree of mixing, so as to avoid “hot spots” where dust and agglomerate deposition can occur (as opposed to the favorable epitaxial
5 deposition).

In another aspect, the addition of hydrogen to the silane may be used to minimize the negative modes of deposition and maximize the epitaxial deposition. In addition to maintaining the correct amount of fluidization required for conventional (hydraulic) fluidized bed reactors, there are reaction chemistry
10 reasons for this hydrogen dilution. Typical hydrogen dilution has a 10-20% silane concentration in the bottom feed gas. In a conventional (hydraulic) fluidized bed reactor, the requirements for fluidization can conflict with the requirements for optimum epitaxy. This can cause the range for optimum operation to be very narrow, and somewhat hard to maintain. With
15 mechanically vibrated fluidized bed reactors and mechanically vibrated packed bed reactors, the hydraulic (i.e., gas flow) needs are uncoupled from those of optimum reaction, which makes control and turndown easier. The reduced need for dilution hydrogen also simplifies the operation.

The height of the bed and the seed particle injection rate are other
20 parameters that must be controlled in mechanically vibrated fluidized and/or packed or compressed bed reactors. Specifically, a long enough retention rate must be provided without having too high or too low of a specific surface area (m2/m3) of particles.

Referring now to mechanically vibrated fluidized bed reactors,
25 loose solid beds (without upper bed constraints) can be made to have a liquid-like motion (similar to conventional hydraulic fluidized bed reactors) by vertically vibrating the bed from below. In this type of bed reactor there is little vertical gas flow. Typically, the only vertical gas flow is from the injection of the minimum reaction gases (i.e., there is limited or no hydrogen dilution other than

the requirements for chemical reaction control). Instead, particles are held in a suspended condition by the rapid up and down motion of a bottom platen, in the order of tens of times per second. The mechanical momentum transfer from the bottom platen to the solids above provides the same counter-balance to the bed weight as in a conventional (gas-hydraulic) fluidized bed reactor. Except for the bottom-most and top-most solids in the bed, each particle's motion is impacted only from the particle(s) immediately below. Each particle then transfers that momentum to the particle(s) immediately above or to the side. Radial movement in a vibrationally fluidized bed reactor is usually significant depending on the degree of particle acceleration.

Typically, mechanically vibrated fluidized bed reactors have characteristic vertical and radial patterns of solids flow, depending on the relative acceleration to gravity (Γ), ($\Gamma = 4\pi^2 f^2 a/g$, where f is the vibration frequency in cycles/sec, a is the vibration amplitude from the neutral point, and g is the gravitational constant). While there are break-points for the solids' flow patterns at various Γ values, there are strong analogies between mechanically vibrated fluidized bed reactors and hydraulically fluidized bed reactors. These analogies include improvements in heat transfer and reaction kinetics.

In a mechanically vibrated fluidized bed reactor, the gas is circulated around the vibrating particles, with the localized gas flow pattern resulting primarily from the localized particle movement, as well as the net pumping action of the bottom platen. While the net bulk gas velocity and net bulk solid particles' velocity are low, their instantaneous velocities are much higher, resulting in improved kinetics and heat transfer. At certain breakpoints of relative acceleration (Γ), there may be shifts in the radial pattern of particles (and therefore gas in each particle's boundary layer), such that the particles move inward at the bottom, rise upward in the middle of the bed, and flow radially outward at the top of the bed. At very high Γ values, there can be localized gas pockets that form and collapse. There are limited analogies

between these breakpoints, and the different flow patterns of conventional (hydraulically) fluidized bed reactors.

Generally, as the Γ value increases (i.e., the bottom platen starts sinusoidal vertical motion) all particles start moving and there is a slight bed
5 expansion from the static condition, but typically just a few percent. When the Γ value is greater than the first breakpoint (about Γ =1.2), the larger particles tend to rise in the bed, and smaller ones tend to fall to the bottom. Below this first breakpoint, there is little mixing of different particle sizes. At very high relative acceleration (above Γ =6), the solids bed becomes well mixed.

10 Referring now to product withdrawal, the product withdrawal from a mechanically vibrated fluidized bed reactor is typically from the unconstrained top, while the product withdrawal is from the bottom in a conventional (hydraulically) fluidized bed reactor. Additionally, the method of controlling fluidization is also different between a hydraulically fluidized bed reactor and a
15 mechanically vibrated fluidized bed reactor. Control feedback is possible with a hydraulically fluidized bed reactor by using the bed's pressure drop. In contrast, a mechanically vibrated fluidized bed reactor has virtually identical pressures at the top and bottom of the bed. Instead, control feedback for fluidization on a mechanically vibrated fluidized bed reactor is performed using
20 accelerometers and phase angle shift sensors.

The above-described differences occur when the top of the vibrated solids bed is mechanically constrained, either totally or partially. When the top of the bed is mechanically constrained, the solids bed acts quite differently in particle and gas movement, expansion, segregation and the
25 importance of relative acceleration. Such a totally or partially constrained bed reactor is referred to herein and in the claims as a mechanically vibrated packed bed reactor or alternatively as a mechanically vibrated compressed bed reactor.

Referring now to semi-constrained mechanically vibrated packed bed reactors, a mechanically vibrated packed bed reactor is an extension of the mechanically vibrated fluidized bed reactors. In this technology, the solids bed is partially constrained at the top, mechanically limiting the bed by one or more horizontal baffles (e.g., a lid or cover above the bed), which cover 80-95% of the bed cross-section. The baffle(s) or lid of the top restraint suppresses radial and vertical solids flow. However, the baffle(s) or lid allow a very modest bed expansion (e.g., about 1% to about 5%) and some minor degree of vertical particle movement. Additionally, there should be enough open cross-sectional area (i.e., non-restrained area) to allow product withdrawal and escape of reaction gases.

The movement of solids in a vibrating fluidized bed reactor at lower acceleration (Γ) is generally initiated at the top, while at higher accelerations (Γ) the movement of solids is generally initiated at the bottom, near the platen. Thus, by limiting the bed so that bed expansion is only a few percent (but with all particles in motion and with surfaces nearly always in contact), a mechanically vibrated fluidized bed reactor may be converted into a mechanically vibrated packed or compressed bed reactor. The degree of constraint is experimentally determined, so as to not mechanically grind the bed particles, over-stress the bed restrainer, or impede the smooth flow of reactant gases. Accordingly, the net movement of a particle is on the order of a few tenths of an inch per minute (a few mm/minute), or a tenth of an inch per second or less. With sufficient acceleration (Γ) the larger particles will still work their way up to the top of the bed, toward the product withdrawal.

In another implementation, the main section of a mechanically vibrated packed or compressed bed reactor may be combined with the product withdrawal section of a mechanically vibrated fluidized bed reactor. For example, a mechanically vibrated packed or compressed bed reactor is similar in some respects to a mechanically vibrated fluidized bed reactor, utilizing the natural segregating effect and solids flow pattern of a mechanically vibrated

fluidized bed reactor at a top center of the mechanically vibrated packed or compressed bed reactor. In the mechanically vibrated packed or compressed bed reactor, growing solid particles move very slowly upward, generally with little net radial particle motion, with the movement controlled by the orientation
5 and placement of the top baffle or lid restraint. In the center of the mechanically vibrated packed or compressed bed reactor, a non-constrained top (*i.e.*, an opening in the lid at the center of the lid) allows that portion of the bed to act like the mechanically vibrated fluidized bed reactor, allowing the product to overflow into a product withdrawal tube like a liquid. Yet for the majority of the
10 bed cross-section, the particle movement is that of a vibrated packed bed reactor. Incoming reaction gases slowly move toward the upper peripheral outlet, with a low net gas velocity, but substantially higher localized velocity relative to the solid particles. The various implementations may include a flange that extends radially from the product withdrawal tube. The product
15 withdrawal tube is referred to interchangeably herein as an overflow tube. The flange may facilitate entry of relative large particles into the product withdrawal tube and/or facilitate in situ classification of particles based on relative size of the particles.

The headings provided herein are for convenience only and do
20 not interpret the scope or meaning of the embodiments.

Figure 1A shows an illustrative crystal production system 1000 that includes one or more systems or subsystems to separate coated particles 22 from a particulate bed 1004 and to charge or re-charge a crucible in a melter 1052 in a high vacuum environment, and thereby suppress dust, according to
25 various implementations.

The particulate bed 1004 can be located in a reactor, for example a mechanically vibrated fluidizing bed reactor or a mechanically vibrated packed or compressed bed reactor. The reactor can include a pan or tray 1018. The pan or tray 1018 can have a surface 1009 which supports and

imparts momentum to the particles 22. The pan or tray 1018 can have a periphery 1020 and one or more peripheral walls 1022 extending about at least a portion of the periphery 1020 to at least partially delimit a retention volume to temporarily retain the particles 22. The reactor includes a least one actuator (e.g., electric motor, solenoid) 60 (Figure 2) coupled (e.g., via a drive train) which is operable to mechanically vibrate the pan or tray 1018 to vibrate or oscillate the particles 22. The actuator vibrates the pan or tray 1018 at least along a vertical axis.

The reactor includes a hollow particle removal tube 132, having an inlet or entrance positioned above an upper surface of the mechanically vibrated particulate bed. In operation, larger particles move upward in the mechanically vibrated particulate bed, exiting the bed through the hollow particle removal tube 132, for eventual delivery to the melter, or coated particle melter, 1050.

The melter, or coated particle melter, 1050 may be used to melt material in order to pull ingots or boules of a material. For example, the melter 1050 can be used to melt a semiconductor material, for instance silicon, to pull ingots or boules of single crystal silicon. Thus the melter 1050 may optionally include one or more crystal production devices 1070 (e.g., puller).

The crystal production system 1000 may include one or more conduits 1030 to convey separated coated particles 1032 from the particulate bed 1004 and to charge or re-charge the crucible 428 in a high vacuum environment. The environment preferably has a low oxygen level or a very low oxygen level and contains low levels of contaminants or very low level of contaminants.

In at least some implementations, a first gaseous chemical species is introduced to the particulate bed 1004. At least a portion of the first gaseous chemical species is decomposed in the particulate bed 1004 to provide a second chemical species which deposits on at least a portion of the

particulates in the particulate bed. The particulates containing the second chemical species provide a plurality of coated particles 22 which, at times, freely circulate throughout the particulate bed 1004. On a periodic, intermittent, or continuous basis, at least a portion of the plurality of coated particles 22 are
5 separated from the particulate bed 1004 and directed to a conduit 1030. The conduit 1030 receives some or all of the separated coated particles 1032.

As used herein, the term “low contaminant level” refers to an environment which favors the production of second chemical species crystals having low contamination levels (e.g., “solar grade” silicon, polysilicon,
10 polycrystalline silicon, or monocrystalline silicon crystals) that meet at least one of the following specifications: an oxygen concentration of less than $1.5x10^{-17}$ atoms per cubic centimeter (atoms/cc); a carbon concentration of less than about $4.5x10^{16}$ atoms/cc; a donor impurities concentration of less than about 7.8 parts per billion atomic (ppba); an acceptor impurities concentration of less
15 than about 2.7 ppba; and total metal impurities (e.g., iron, chrome, nickel, copper, zinc) of less than about 0.2 parts per million by weight (ppmw), or of less than about 50 parts per billion by weight (ppbw), or of less than about 10 parts per billion by weight (ppbw) .

As used herein, the term “very low contaminant level” refers to an
20 environment which favors the production of second chemical species crystals having very low contamination levels (e.g., “electronics grade” silicon, polysilicon, polycrystalline silicon, or monocrystalline silicon crystals) that meet or exceed at least one of the following specifications: an oxygen concentration of less than $1.0x10^{-17}$ atoms per cubic centimeter (atoms/cc); a carbon
25 concentration of less than about 80 ppba; a donor (e.g., phosphorous, arsenic, antimony) impurities concentration of less than about 150 parts per trillion atomic (ppta); an acceptor (e.g., boron, aluminum) impurities concentration of less than about 50 ppta; bulk metal impurities (e.g., iron, chrome, nickel, copper, zinc) of less than about 1.5 parts per billion by weight (ppbw); surface
30 iron concentration of less than about 2 ppbw; surface copper concentration of

less than about 500 parts per trillion by weight (pptw); surface nickel concentration of less than 500 pptw; surface chromium concentration of less than 500 pptw; surface zinc concentration of less than 1000 pptw; and surface sodium concentration of less than about 2000 pptw.

5 The systems and methods described herein are applicable to a variety of different crystal production methods. For example, all or a portion of the separated coated particles 1032 may be introduced to a Czochralski production process in which a second chemical species crystal (*e.g.*, a crystal formed by the separated coated particles 1032) is progressively melted and 10 solidified to provide a second chemical species crystal having a high purity. In another embodiment, all or a portion of the separated coated particles 1032 may be introduced to a Bridgman-Stockbarger crystal production process in which a crucible containing the molten second chemical species is cooled at a controlled rate to produce a second chemical species crystal having a high 15 purity, or alternatively in another process, for instance a Float Zone crystal production process.

At times, the conduit 1030 is a simple transport device or system capable of moving, transporting, or otherwise conveying at least a first portion of separated coated particles 1034 to the coated particle melter 1050. At other 20 times, the conduit 1030 can include multiple unit operations, such as separated coated particle storage/accumulation, separated coated particle size classification, and/or separated coated particle size reduction processes. Regardless of the functions provided by the conduit 1030, at all times the conduit 1030 maintains the separated coated particles 1032 in an environment 25 having an environment that is maintained at a low oxygen level or a very low oxygen level. Such low oxygen environments advantageously minimize, reduce or even eliminate oxide formation on the surface of the separated coated particles 1032.

At times, the conduit 1030 can include one or more apparatuses, 30 systems, or devices that are hermetically sealed to and fluidly couple one or

more fluidized bed coated particle production processes to one or more crystal production systems or devices, such as one or more coated particle melters 1050 and crystal production devices 1070. At other times, the conduit 1030 can include one or more moveable apparatuses, systems, or devices that are
5 capable of being hermetically sealed and fluidly coupled to one or more fluidized bed coated particle production processes and hermetically sealed and fluidly coupled to one or more crystal production systems or devices, such as one or more coated particle melters 1050 and crystal production devices 1070.

10 The minimization, reduction, or elimination of oxide formation on the surface of the separated coated particles 1032 is particularly advantageous when the second chemical species includes silicon, since the formation of silicon oxides can significantly compromise the purity and/or quality of silicon crystals produced using the separated silicon coated particles 1032 and
15 disadvantageously make it more difficult to melt the smaller diameter silicon coated particles. The presence of silicon oxides on the surface of silicon coated particles detrimentally increases the melt time and energy required to melt such particles when compared to silicon coated particles without a silicon oxide layer. At times, it is believed the presence of silicon oxides on the
20 surface of silicon coated particles 1032 may have melting points that exceed the melting point of pure silicon (i.e., silicon coated particles lacking the silicon dioxide layer) by at least about 10°C; at least about 50°C; or at least about 100°C.

Such effects are particularly evident with smaller diameter silicon
25 coated particles because – while the thickness of the silicon oxide shell is independent of the silicon coated particle diameter (e.g., the shell may be from 10 to 20 silicon dioxide molecules thick) – the mass ratio of the silicon dioxide layer on the surface of the silicon coated particle to the mass of the particle is inversely proportional to the diameter of the particle. For example, when the
30 diameter is reduced by one-half the aforementioned mass ratio of silicon

dioxide on the surface of a smaller diameter silicon coated particle to pure silicon in the interior of the smaller diameter silicon coated particle increases 2 times.

At times, the particulate bed 1004 can be disposed at least partially in a reactor housing 1002 defining a chamber. A flexible member 42 may apportion the chamber into an upper chamber 1003 and a lower chamber 1003A. The upper chamber 1003 may be maintained at one or more defined temperatures or temperature ranges. The temperature of the particulate bed 1004 may be altered, adjusted, or controlled using one or more thermal energy emitting systems 1008, for example one or more electric resistance heaters or one or more heat transfer surfaces that uses a circulated thermal transfer fluid (e.g., thermal oil) or material (e.g., molten salt). At times, the temperature of the particulate bed 1004 can be controlled to exceed the thermal decomposition temperature of the first gaseous chemical species while the temperature at other points in the upper chamber 1003 can be controlled to lower than the thermal decomposition temperature of the first gaseous chemical species. In some implementations, one or more thermal energy transfer devices 1012 may be physically and/or thermally conductively coupled to the vessel 1002 to remove thermal energy (i.e., heat) from the upper chamber 1003. Additionally or alternatively, one or more of the elements or functions explained above or below with regard to the upper chamber 1003 may create or apply in the lower chamber 1003A an environment or a condition that is the same as or similar to one or more of the above or below explained environments or conditions of upper chamber 1003. Such may advantageously assist in maintaining or creating one or more of the above or below explained environments or conditions of the upper chamber 1003.

The thermal energy emitting systems 1008 increase the temperature of the particulate bed 1004 above the thermal decomposition temperature of the first gaseous chemical species. For example, where the first gaseous chemical species includes silane, the thermal energy emitting systems

1008 can increase the temperature of the particulate bed 1004 above 420°C, the onset thermal decomposition temperature of silane. In at least some implementations, the first gaseous chemical species may be preheated prior to introduction to the particulate bed 1004. Preheating of the first gaseous chemical species beneficially reduces the heat load on the thermal energy emitting systems 1008. The first gaseous chemical species may be preheated to about 100°C; about 200°C; about 300°C; or about 400°C. The first gaseous chemical species may be heated using a feed heater or a heat interchanger where hot gases leaving the particulate bed 1004 are used as the pre-heating medium. The thermal energy emitting systems 1008 may include any number or combination of thermal energy emitting devices, systems, or combinations thereof. The thermal energy emitting systems 1008 can increase the temperature of the surface 1009 supporting the particulate bed 1004, thereby conductively transferring heat to and raising the temperature of the particulate bed 1004. The thermal energy emitting devices 1008 can include any number or combination of electrically powered heating elements such as resistive heaters (e.g., Calrod, Nichrome, and the like), ceramic heating elements (e.g., molybdenum disicilide, PTC ceramics, and the like), and/or radiant heating elements positioned beneath and proximate the surface 1009 supporting the particulate bed 1004. The thermal energy emitting devices 1008 can also include any number or combination of circulated heat transfer fluid systems, for example Dynalene molten salts (Dynalene, Inc. Whitehall, PA).

At times, the first gaseous chemical species may be heated to a temperature in the range of from about 50°C to about 450°C, or about 350°C. Preheating the first gaseous chemical species to a temperature of about 350°C beneficially reduces the heat load on the thermal energy emitting systems 1008. The thermal energy used to raise the temperature of the first gaseous chemical species can be supplied in whole or in part using one or more external electric heaters. Such thermal energy may be provided by one or more external electric heaters, one or more external fluid heaters, or one or more

heat interchanges or exchangers where hot gases are used to heat the incoming feed.

Passing at least a portion of the first gaseous chemical species through the upper zone of the reactor housing 1002 may preheat the first gaseous chemical species to a temperature that is below the thermal decomposition temperature of the first gaseous chemical species. At times, the first gaseous chemical species may be passed through one or more heat exchange stages located in the upper chamber 1003 of the reactor housing 1002 where the temperature of the first gaseous chemical species is increased to a level that is slightly less than the thermal decomposition temperature of the first gaseous chemical species. Further, the temperature of the gas in upper chamber 1003 may be controlled below the decomposition temperature of the first gaseous chemical species by means of auxiliary cooling (e.g., a fluid cooler in a cooling coil) positioned in the upper chamber or upper zone of the chamber 1003. Such an approach provides several benefits:

1. The mixed first gaseous chemical species to the particulate bed 1004 is controlled at an optimum temperature; and
2. The upper zone of the chamber 1003 in the reactor housing 1002 is maintained below the decomposition temperature, minimizing, reducing or even eliminating the thermal decomposition of the first gaseous chemical species at locations in the reactor housing 1002 external to the particulate bed 1004.

The reactor housing 1002 can include one or more thermal energy transfer systems 1012 that maintain the temperature of the upper chamber 1003 below the thermal decomposition temperature of the first gaseous chemical species. Maintaining the temperature of the upper chamber 1003 below the thermal decomposition temperature of the first gaseous chemical species advantageously reduces the likelihood of the first gaseous chemical species decomposing in the upper chamber 1003 in locations external to the particulate bed 1004. In other words, maintaining the temperature of the

particulate bed 1004 above the thermal decomposition temperature of the first gaseous chemical species while maintaining the temperature elsewhere in the upper chamber 1003 below the thermal decomposition temperature of the first gaseous chemical species beneficially favors deposition of the second chemical species in the particulate bed 1004 rather than on surfaces in the upper chamber 1003. In some implementations, the first gaseous chemical species is maintained at a temperature below the thermal decomposition temperature of the first gaseous chemical species at all times and locations prior to discharge into the particulate bed 1004. The one or more thermal energy transfer systems 1012 can include any number or combination of systems and/or devices suitable for maintaining the upper chamber 1003 at a temperature below the thermal decomposition temperature of the first gaseous chemical species, including internal cooling coils.

The exhaust gas system 1010 is fluidly coupled to the upper chamber 1003 to receive exhaust gasses from the upper chamber 1003. The decomposition of the first gaseous chemical species in the particulate bed 1004 may, at times, produce one or more inert byproducts, for example one or more third gaseous chemical species. Left in the upper chamber 1003, such gaseous byproducts can accumulate and adversely affect system pressure control and the conversion and/or yield of the first gaseous chemical species to the second chemical species. To limit their accumulation in the upper chamber 1003, gaseous byproducts are removed via one or more exhaust gas systems 1010.

A first gaseous chemical species feed system 1006 supplies the first gaseous chemical species to the particulate bed 1004. The first gaseous chemical species feed system 1006 can include one or more first gaseous chemical species reservoirs for storing the first gaseous chemical species, a distribution header 350, and any number of injectors 356 fluidly coupled to the distribution header 350 and positioned in the particulate bed 1004. In some instances, the distribution header 350 and/or the number of injectors 356 can

be thermally insulated to limit heating of the first gaseous chemical species in the distribution header 350 and/or injectors 356. In such instances, the thermal insulation may limit the temperature of the first gaseous chemical species in the distribution header 350 and/or injectors 356 to less than the thermal
5 decomposition temperature of the first gaseous chemical species.

In some instances, a dopant feed system 1014 supplies one or more dopants to the upper chamber 1003 or directly to the particulate bed 1004. At times, the dopant feed system 1014 is fluidly coupled to the first gaseous chemical species feed system 1006 such that the first gaseous
10 chemical species and the dopant are supplied via the number of injectors 356 to the particulate bed 1004. At other times, the dopant feed system 1014 is separately fluidly coupled to the particulate bed 1004 and/or the upper chamber 1003. The one or more dopants can be added to the particulate bed 1004 contemporaneous with the feed of the first gaseous chemical species to the
15 particulate bed 1004 to produce doped coated particles. Additionally or alternatively, the one or more dopants may be added to the particulate bed 1004 at times when the first gaseous chemical species is not added to the particulate bed 1004. Illustrative dopants may include, but are not limited to, arsenic, germanium, selenium, and/or gallium. An example doped coated
20 particle 22 produced by the reactor system 1000 includes coated particles 22 containing boron or phosphorous doped silicon. Dopant may depend on the crystallization technique. A CZ process will typically take solids in the form of predoped and crushed silicon (B and P doped), or can even employ elemental Boron or white Phosphor. Gas dopants may include Phosphine and Diborane.
25 The decomposition of the first gaseous chemical species can include one or more chemical decomposition processes, one or more thermal decomposition processes, or combinations thereof. For example, the first gaseous chemical species can include a silicon-containing gas that thermally decomposes when introduced to a heated particulate bed 1004 held at a
30 temperature in excess of the thermal decomposition temperature of the first

gaseous chemical species. The nonvolatile second chemical species is produced by the decomposition of the first gaseous chemical species and may deposit on proximate surfaces (*e.g.*, the surfaces of the particulates in the particulate bed 1004) at the moment of decomposition of the first gaseous
5 chemical species. At times, the first gaseous chemical species can include a silicon containing gas and the second chemical species can include silicon. Non-limiting examples of such silicon containing gases include silane (SiH_4); dichlorosilane (H_2SiCl_2); or trichlorosilane ($HSiCl_3$). At times one or more byproduct third gaseous species (e.g., hydrogen, hydrogen chloride) may be
10 generated by the thermal decomposition of the first gaseous chemical species in the particulate bed 1004.

The first gaseous chemical species may be supplied to the particulate bed 1004 via the one or more injectors 356, each of which includes one or more outlets positioned in the particulate bed 1004 such that the first
15 gaseous chemical species travels at least a minimum defined distance through the particulate bed 1004 or, alternatively, is retained in the particulate bed 1004 for at least a defined minimum retention time. In addition to depositing the non-volatile second chemical species on at least some of the particulates in the particulate bed 1004, the decomposition of the first gaseous chemical species
20 can produce one or more third gaseous byproducts, such byproducts are typically inert and may, at times, be chemically similar or identical to the one or more diluents used to adjust the concentration of the first gaseous chemical species in the particulate bed 1004.

Coated particles 22 are separated from the particulate bed 1004
25 using any current or future developed separation system or process including mechanical or hydraulic fluidization of the particulate bed 1004 coupled with one or more devices or systems capable of selectively separating the coated particles 22 from the particulate bed 1004. Coated particles 22 removed from the particulate bed 1004 (i.e., separated coated particles 1032) are collected
30 and directed to the conduit 1030. At times, the separated coated particles 1032

can have a dp_{50} (i.e., the mass of coated particles smaller than the specified size comprising 50% of the total sample) less than or equal to 10000 micrometers (µm); less than or equal to 5000 µm; less than or equal to 3000 µm; less than or equal to 2000 µm; less than or equal to 1000 µm; less than or equal to 500 µm; less than or equal to 300 µm; less than or equal to 100 µm. At times, the separated coated particles 1032 can have a diameter of about 10 micrometers (µm) or more; about 20 µm or more; about 50 µm or more; about 100 µm or more; about 200 µm or more; about 500 µm or more; or about 1000 µm or more.

At times, the separated coated particles 1032 may have a Gaussian particle size distribution with a minimum size of less than about 10 micrometers (µm); less than about 20 µm; less than about 50 µm; less than about 75 µm; less than about 125 µm; less than about 150 µm; or less than about 200 µm. At times, the separated coated particles 1032 may have a Gaussian particle size distribution with a maximum size of less than about 300 micrometers (µm); less than about 500 µm; less than about 600 µm; less than about 750 µm; less than about 1 millimeter (mm); less than about 1.5 mm; less than about 2 mm; or less than about 5 mm. At times, the separated coated particles 1032 may have a Gaussian particle size distribution with a mean size of about 100 micrometers (µm); about 200 µm; about 400 µm; about 600 µm; about 800 µm; about 1 millimeter (mm); about 1.5 mm; or about 2 mm.

The environment within the upper chamber 1003 is maintained at a low oxygen level (e.g., less than 20 volume percent oxygen) or a very low oxygen level (e.g., less than 0.001 mole percent oxygen to less than 1 mole percent oxygen). In some instances, the environment within the upper chamber 1003 is maintained at a low oxygen level that does not expose the coated particles 22 to atmospheric oxygen. In some instances, the environment within the upper chamber 1003 is maintained at a low oxygen level having an oxygen concentration of less than 20 volume percent (vol%). In some instances, the environment within the upper chamber 1003 is maintained

at a very low oxygen level having an oxygen concentration of less than about 1 mole% (mol%); less than about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol%; less than about 0.001 mol%.; or less than about 0.0000001 mol%.

5 Since the upper chamber 1003 is maintained at a low oxygen level or a very low oxygen level, oxide formation on the surface of the coated particles 22 is beneficially minimized, reduced, or even eliminated. In one example, silicon coated particles 22 produced in the heated particulate bed 1004 can have an oxygen content as silicon dioxide of less than about 100
10 parts per million atomic (ppma) oxygen; less than about 50 ppma oxygen; less than about 10 ppma oxygen; less than about 1 ppma oxygen; or loss than 1 part per billion atomic (ppba) oxygen.

Additionally, since very low levels of contamination exist in the upper chamber 1003 by virtue of the closed environment therein, and since the
15 opportunity for contamination of the coated particles 22 by impurities is minimized by the low or very low oxygen levels and the low or very low contaminant levels, for example metal atoms or ions, in the environment provided by the enclosed conduit 1030, coated particle melter 1050, and crystal production device 1070, the production of second chemical species crystals
20 having very low levels of contamination is possible.

The relatively low levels of contamination achievable in such a production and conveyance process facilitate the use of both small and large diameter separated coated particles 1032 in subsequent crystal production processes. Providing the capability to use small and large diameter coated
25 particles for crystal production can advantageously, at times, eliminate the need to classify and remove smaller diameter coated particles via classification – a process that frequently exposes the separated coated particles 1032 to significant contaminants (e.g., metallic contamination from classification screens) and oxygen.

At times, the upper chamber 1003 is maintained at a low contaminant level environment or a very low contaminant level environment. In some instances, the second chemical species crystals produced using silicon coated particles 22 produced in such low contaminant level or very low contaminant level environments can meet or exceed electronics grade silicon specifications. In such instances, the second chemical species crystals produced by the crystal production device 1070 can have a resistivity of greater than about 250 Ohm-centimeters (Ω-cm); an oxygen concentration of less than $1.0x10^{-17}$ atoms per cubic centimeter (atoms/cc); a carbon concentration of less than about 80 ppba; a donor (e.g., phosphorous, arsenic, antimony) impurities concentration of less than about 150 parts per trillion atomic (ppta); an acceptor (e.g., boron, aluminum) impurities concentration of less than about 50 ppta; bulk metal impurities (e.g., iron, chrome, nickel, copper, zinc) of less than about 1.5 parts per billion by weight (ppbw); surface iron concentration of less than about 2 ppbw; surface copper concentration of less than about 500 pptw; surface nickel concentration of less than 500 pptw; surface chromium concentration of less than 500 pptw; surface zinc concentration of less than 1000 pptw; and surface sodium concentration of less than about 2000 pptw.

In other instances, the second chemical species crystals produced using silicon coated particles 22 produced in such low contaminant level or very low contaminant level environments can meet or exceed solar grade silicon specifications. In such instances, the second chemical species crystals produced by the crystal production device 1070 can have a resistivity of greater than about 20 Ohm-centimeters (Ω-cm); an oxygen concentration of less than $1.5x10^{-17}$ atoms per cubic centimeter (atoms/cc); a carbon concentration of less than about $4.5x10^{16}$ atoms/cc; a donor impurities concentration of less than about 7.8 parts per billion atomic (ppba); an acceptor impurities concentration of less than about 2.7 ppba; and total metal impurities (e.g., iron, chrome, nickel, copper, zinc) of less than about 0.2 parts per million by weight (ppmw).

As the second chemical species deposits on the particulates in the particulate bed 1004, the diameter of the coated particles 22 present in the particulate bed 1004 increases. In some instances, the second chemical species can deposit on the surface of the particulates and coated particles present in the particulate bed 1004 in the form of sub-particles, thereby forming coated particles comprising an agglomeration of smaller second chemical species sub-particles. In some instances, the second chemical species can deposit in layers on the surface of the particulates and coated particles present in the particulate bed 1004. Coated particles 22 meeting one or more physical and/or compositional criteria are separated from the particulate bed 1004. In some instances, the separated coated particles 1032 travel through a hollow particle removal tube 132 and are deposited in the conduit 1030.

At times, the conduit 1030 can be as simple as a hollow tube that connects and hermetically seals the upper chamber 1003 in the reactor 1002 to the coated particle melter 1050. At other times, the conduit 1030 can include a number of individual unit operations that includes, but is not limited to, one or more of the following: coated particle 1032 storage or accumulation; separated coated particle 1032 size classification; separated coated particle 1032 apportioning into at least a first portion of separated coated particles 1034 and a second portion of separated coated particles 1038; or separated coated particle 1032 size reduction.

At times, the conduit 1030 may include one or more fixed components, devices, and/or systems that fluidly couple and hermetically seal the upper chamber 1003 in the reactor 1002 to the coated particle melter 1050. At other times, the conduit 1030 can include one or more mobile or moveable components, devices, and/or systems that fluidly couple and hermetically seal the conduit 1030 to the upper chamber 1003 in the reactor to receive the separated coated particles 1032, and fluidly couple and hermetically seal the conduit 1030 to the coated particle melter 1050. Regardless of the form of the conduit 1030, the conduit 1030 maintains the separated coated particles 1032

in an environment having a low oxygen level (*e.g.*, less than 20 volume percent oxygen) or a very low oxygen level (e.g., less than 1 mole percent oxygen) that limits the exposure of the separated coated particles 1032 to oxygen.

In some instances, the conduit 1030 can transport a first portion of separated coated particles 1034 to the coated particle melter 1050 in a low oxygen level environment that does not expose the first portion of separated coated particles 1034 to atmospheric oxygen. In some instances, the conduit 1030 can transport the first portion of separated coated particles 1034 to the coated particle melter 1050 in an environment maintained at a low oxygen level having an oxygen concentration of less than 20 volume percent (vol%). In some instances, the conduit 1030 can transport the first portion of separated coated particles 1034 to the coated particle melter 1050 in an environment maintained at a very low oxygen level having an oxygen concentration of less than about 1 mole% (mol%); less than about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol%; less than about 0.001 mol%; or less than about 0.0000001 mol%.

An oxide layer can form on some or all of the exposed surfaces of the separated coated particles 1032 when the particles are exposed to an oxygen-containing environment. In one example, a silicon dioxide layer can form on the surface of separated silicon coated particles 1032. At times, the oxide layer may partially or completely coat or encase the separated coated particles 1032. At times, such oxide layers may be from 10 to 30 silicon dioxide molecules thick. The formation of an oxide layer about the separated coated particles 1032 detrimentally impacts the quality of items produced using the separated coated particles 1032. For example, it can increase the concentration of oxygen in the second chemical species crystals that are produced using the first portion of separated coated particles 1034. In addition, the presence of a silicon dioxide coating across at least a portion of the surface of the first portion of separated coated particles 1034 elevates the *apparent* melting point of the *coated particles*, this elevation is particularly noticeable in

smaller diameter coated particles. For example, the melting point of silicon is approximately 1414°C and the melting point of silicon dioxide is approximately 1700°C. Small diameter separated coated particles 1032 having 10 to 30 molecule thick layer of silicon dioxide may melt less readily at the melting point
5 of pure silicon, detrimentally increasing the melt time and energy required to melt such particles when compared to silicon coated particles without a silicon oxide layer.

Additionally, smaller diameter coated particles (e.g., coated particles having a diameter of less than 100 micrometers to about 500
10 micrometers) tend to “float” on the surface of the molten second chemical species, such as the molten second chemical species pool 1060 present in the coated particle melter 1050. The propensity for such smaller diameter coated particles to float frequently makes it difficult to melt these particles particularly when such smaller diameter coated particles include an oxide layer (e.g., silicon
15 dioxide) that increases the effective melting point of such smaller coated particles coated particles above the melting point of the second chemical species (e.g., pure silicon).

The physical aspects (e.g., size, density, surface area/mass ratio, and the like) of the separated coated particles 1032 can form a distribution. In
20 one implementation, diameters of the separated coated particles 1032 can form a distribution (e.g., a Gaussian distribution) about a mean coated particle diameter or median coated particle diameter. In some implementations, the separated coated particles 1032 in the conduit 1030 can be further classified into a first portion of separated coated particles 1034 forwarded to the coated
25 particle melter 1050 and a second portion of separated coated particles 1038, at least a portion of which are recycled to the particulate bed 1004 via, for example, a hollow particle recycle tube 406. In some instances, the physical aspects (e.g., size, density, surface area/mass ratio, and the like) of the first portion of separated coated particles 1034 can form a first distribution. For
30 example, the respective diameters of coated particles in the first portion of

separated coated particles 1034 can form a Gaussian distribution about a first mean coated particle diameter or a first median coated particle diameter. In some instances, the respective physical aspects (e.g., size, density, surface area/mass ratio, and the like) of coated particles in the second portion of separated coated particles 1038 can form a second distribution. For example, the respective diameters of coated particles in the second portion of separated coated particles 1038 can form a first Gaussian distribution about a second mean coated particle diameter or a second median particle diameter. In some instances, some or all of the first distribution and the second distribution may at least partially overlap. In other instances, the first distribution and the second distribution may not overlap.

At times, the first portion of separated coated particles 1034 can include coated particles having one or more desirable physical or compositional properties or characteristics. Such desirable properties or characteristics may, for example, favor melting the first portion of separated coated particles 1034 in the coated particle melter 1050. For example, the mean diameter of coated particles in the first portion of separated coated particles 1034 may be greater than the mean diameter of coated particles in the second portion of separated coated particles 1038. In some instances, the first portion of separated coated particles 1034 may include coated particles having a mean or a median diameter of greater than about 10 micrometers (μm); greater than about 20 μm; greater than about 50 μm; greater than about 100 μm; greater than about 200 μm; greater than about 300 μm; greater than about 400 μm; greater than about 500 μm; or greater than about 600 μm. In some instances, the first portion of separated coated particles 1034 may include coated particles having a diameter of greater than about 50 micrometers (μm); greater than about 100 μm; greater than about 200 μm; greater than about 300 μm; greater than about 400 μm; greater than about 500 μm; or greater than about 600 μm. The first portion of separated coated particles 1034 can include oxygen as a metallic oxide of less than: about 6000 parts per billion atomic (ppba); less than about

3000 ppba; less than about 1000 ppba; less than about 600 ppba; less than about 250 ppba; less than about 100 ppba; less than about 50 ppba; less than about 20 ppba; less than about 10 ppba; less than about 5 ppba; less than about 1 ppba; less than about 0.5 ppba; less than about 0.1 ppba. It is believed the lower levels of silicon oxides (e.g., silicon dioxide) present on the exposed surfaces of the first portion of separated coated particles 1034 advantageously enables the use of smaller diameter coated particles in the production of second chemical species crystals.

At times, the second portion of separated coated particles 1038 can include coated particles having one or more desirable physical or compositional properties or characteristics. Such desirable physical or compositional properties or characteristics may, for example, favor returning some or all of the second portion of separated coated particles 1038 back to the particulate bed 1004 and/or removing some or all of the second portion of separated coated particles 1038 from the crystal production system 1000 for additional processing. Such additional processing may include, for example, physically reducing the size (*e.g.*, via grinding) some or all of the second portion of separated coated particles 1038 to a smaller diameter for use as “start-up” or seed particulate returned to the particulate bed 1004. In some instances, the second portion of separated coated particles 1038 may include coated particles having a mean or a median diameter of less than about 600 micrometers (μm); less than about 500 μm; less than about 400 μm; less than about 300 μm; less than about 200 μm; less than about 100 μm; or less than about 50 μm. In some instances, the second portion of separated coated particles 1038 may include coated particles having a diameter of less than about 600 micrometers (μm); less than about 500 μm; less than about 400 μm; less than about 300 μm; less than about 200 μm; less than about 100 μm; or less than about 50 μm.

Coated particles such as the separated coated particles 1032 can be classified by physical or compositional properties or characteristics in any of several locations. At times, such classification may be performed in one or

more unit operations in the conduit 1030. In one implementation, the separated coated particles 1032 may be classified by physical or compositional properties or characteristics as the coated particles 22 are separated from the particulate bed 1004 in the reactor housing 1002. In another implementation, the separated coated particles 1032 may be classified by physical or compositional properties upon selective separation into the first portion of separated coated particles 1034 and the second portion of separated coated particles 1038 in one or more unit operations in the conduit 1030. By physically or compositionally classifying coated particles, such as the separated coated particles 1032, in low oxygen level or very low oxygen level environments such as in the reactor 1002 or in the conduit 1030, oxide formation on the external surface of the separated coated particles 1032 is minimized, reduced, or even eliminated.

By providing a hermetic seal, low oxygen level environment, very low oxygen level environment, or oxygen free environment between the reactor 1002 and the coated particle melter 1050, the conduit 1030 beneficially and advantageously minimizes, reduces, or even eliminates oxide formation on the exposed surfaces of the separated coated particles 1032. Taking the separated silicon coated particles 1032 as an illustrative example; the elimination of an oxide layer (e.g., silicon oxide, silicon dioxide) on the exposed surfaces of the separated coated particles 1032 provides numerous benefits and advantages over systems and methods in which the separated coated particles 1032 are unavoidably and/or inadvertently exposed to elevated or atmospheric oxygen levels, such as during handling, storage, and/or transfer.

One such advantage is small diameter separated coated particles can be included in the first portion of separated coated particles 1034 forwarded to the coated particle melter 1050. Smaller diameter separated silicon coated particles traditionally have been excluded from coated particle melters due to difficulties in melting the smaller particles (e.g., causing a possible melting point rise associated with a higher ratio of mass of silicon dioxide in a shell to mass of silicon inside such silicon dioxide shell; dusting issues inside of coated

particle melters; and small diameter coated particles floating on a surface of a molten second chemical species in coated particle melters due to low density) and the detrimental effect on quality of silicon crystals pulled from melted silicon (e.g., due to oxygen contamination of pulled silicon crystal) caused by an oxide layer carried by small sized particles fed to coated particle melters — because the ratio of oxygen in small particles to particle mass is proportionately greater in small particles than in larger particles. Consequently, the separated coated particle size distribution of the first portion of separated coated particles 1034 can include smaller diameter coated particles, thereby reducing or even eliminating the need for coated particle classification (and potential for subsequent oxygen exposure) in the conduit 1030. Eliminating classification advantageously eliminates a size classification unit operation and the attendant handling of coated particles upstream and downstream of the classification unit operation. Such elimination reduces or eliminates introduction of contaminants (e.g., oxygen, atomic metals, metallic particulates, and others) to the separated coated particles 1032 that may otherwise occur during such size classification unit operation and attendant handling of coated particles upstream and downstream of the classification unit operation. Further, the ability to feed a wide range of coated particle sizes to the coated particle melter 1050 increases the density of the crucible pack because void spaces within the crucible 428 are reduced.

Dusting (suspension of fine coated particles) in the coated particle melter 1050 has been a problem solved by removing small diameter coated particles from the feed to coated particle melter 1050. Advantageously, small diameter coated particles produced in a mechanically vibrated packed particulate bed 1004 have a different morphology and density than coated particles produced using a hydraulically fluidized bed. Small diameter coated particles produced in the mechanically vibrated particulate bed 1004 and/or a mechanically fluidized particulate bed 20 are believed to be more spherical in shape and, consequently, are believed to have a higher density than small

diameter particles produced in a legacy hydraulically fluidized bed. The density of the small diameter particles contained in separated coated particles 1032 from particulate beds 1004 and/or mechanically fluidized beds 20 may be 10 to 100 times greater than the density of coated particles produced in a
5 hydraulically fluidized bed.

Such small diameter separated coated particles 1032 can measure smaller than 400 micrometers, smaller than 300 micrometers, smaller than 200 micrometers, smaller than 100 micrometers, smaller than 500 micrometers, and smaller than 10 micrometers.

10 For example, the bulk density of smaller diameter coated particles produced in a particulate bed 1004 and/or a mechanically fluidized particulate bed 20 may be 1 gram per cubic centimeter. In contrast, the density of smaller diameter coated particles produced in a hydraulically fluidized bed may be as low as 0.01 to 0.1 grams per cubic centimeter. Aerodynamic sphericity of
15 coated particles 22 produced in a particulate bed 1004 and/or a mechanically fluidized particulate bed 20 approaches 0.98 compared to an aerodynamic sphericity of 0.5 to 0.6 for coated particles produced in a hydraulically fluidized bed. Due to these differences, small diameter coated particles produced in the mechanically fluidized bed 1004 tend to cause fewer observable dusting
20 problems in the crystal production operation. In addition, the small diameter particles contained in coated particles 22 generated in the mechanically fluidized particulate bed 20 have differing physical properties and/or morphological properties from small diameter coated particles produced using a hydraulically fluidized bed. For example, it is believed that small diameter
25 particles contained in coated particles 22 produced in the mechanically fluidized particulate bed 20 may be less “sticky” (i.e., may demonstrate lower tendency to adhere to each other and to surfaces) than small diameter coated particles generated using a hydraulically fluidized bed. Stated differently, small diameter coated particles produced in a hydraulically fluidized particulate have a
30 propensity – likely related to a unique surface chemistry and/or morphology – to

cling to surfaces and not flow smoothly. In contrast, small diameter particles contained in coated particles 22 produced in the mechanically fluidized bed 20 tend to demonstrate a lower propensity to cling and have a greater tendency to flow smoothly throughout the production, conveyance, and crystal production processes. In another example, it is believed that the physical structure of small diameter particles contained in coated particles 22 produced in the mechanically fluidized bed reactor may have greater density and/or may be more spherical than small diameter particles produced in hydraulically fluidized beds. These physical properties make coated particles 22, including smaller diameter coated particles, produced in the mechanically fluidized bed reactor more amenable to melting in a crystal production process.

The coated particle melter 1050 can include any system, device, or combination of systems and devices to heat the first portion of separated coated particles 1034 to a temperature at or above the melting point of the second chemical species and provide a crucible 428 that contains the molten second chemical species pool 1060. At times, the coated particle melter 1050 maintains a thermal profile along the depth of the reservoir or pool of molten second chemical species. The coated particle melter 1050 may form a portion of or may, alternatively, be replaced by one or more crystal production devices 1070. Such crystal production devices may include, but are not limited to, any current or future developed crystal production device amenable for the production of monocrystalline second chemical species (e.g., monocrystalline silicon). Examples of such crystal production devices include crystal pullers, float-zone crystal production devices, Czochralski crystal production devices, and Bridgman-Stockbarger crystal production devices.

Using silicon as an illustrative second chemical species – silicon expands on crystallization and contracts upon melting. Silicon coated particles are covered in crystalline silicon. When an oxide layer (*e.g.*, silicon dioxide) forms on the external surfaces of silicon coated particles or separated silicon coated particles, without being bound by theory, it is hypothesized that the

oxide layer can act as a microns thick, relatively impermeable, shell surrounding a crystalline silicon coated particle or crystalline silicon coated separated coated particle. When such silicon dioxide separated coated particles are included in the first portion of separated coated particles 1034 and heated during a silicon crystal production process, it is possible for the crystalline silicon to melt while the silicon dioxide shell (which has a melting temperature hundreds of degrees Celsius higher than the melting point of silicon) remains intact. In such instances, it is theorized that the contraction of the molten silicon within the silicon dioxide shell creates a vacuum within the silicon dioxide layer that exerts a compressive or implosive force on the silicon dioxide layer.

It is hypothesized that smaller diameter silicon coated particles are able to better withstand the resultant compressive force better than larger diameter silicon coated particles and, as a result, tend to float and are difficult to melt, requiring a significantly greater energy input to melt small diameter silicon coated particles when such particles are included in the first portion of the separated coated particles 1034. Consequently, it is believed that by minimizing, reducing, or eliminating the formation of silicon dioxide on the external surfaces of the separated coated particles 1032, the “meltability” of the separated coated particles 1032 will be improved and the isolation of small diameter silicon coated particles from the first portion of separated coated particles 1034 is significantly reduced or even eliminated. Since smaller diameter silicon coated particles may be included in the first portion of separated coated particles 1034 used in crystal production, both capital and operating expenses associated with coated particle classifiers or similar separation devices are beneficially reduced or even eliminated. Additionally, by reducing or eliminating the classification of the separated coated particles 1032, the potential for contamination of the separated coated particles 1032 is reduced, as explained above.

Figure 1B shows an illustrative conduit 1030 that includes only a hermetic coupling between the reactor 30 and the coated particle melter 1050, according to one implementation. Such a configuration may be alternatively referred to as a “close-coupled” configuration. In such a configuration, the separated coated particles 1032 are transferred directly to the coated particle melter 1050 via the conduit 1030. In some implementations, the coated particle melter 1050 may have internal ambient or elevated temperature coated particle storage. At times, the environment in the coated particle storage can be maintained at a low oxygen level having an oxygen concentration of less than 20 volume percent (vol%). At other times, the environment in the coated particle storage can be maintained at a very low oxygen level having an oxygen concentration of less than about 1 mole% (mol%); less than about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol%; less than about 0.001 mol%; or less than about 0.0000001 mol%.

Figure 1C shows an illustrative alternative conduit 1030 that includes a coated particle accumulator 1080, according to an embodiment. The separated coated particles 1032 are directed to the coated particle accumulator 1080. The separated coated particles 1032 are transferred from the coated particle accumulator 1080 to the coated particle melter 1050 on demand, intermittently, periodically, or continuously.

At times, the environment in the coated particle accumulator 1080 can be maintained at a low oxygen level having an oxygen concentration of less than 20 volume percent (vol%). At other times, the environment in the coated particle accumulator 1080 can be maintained at a very low oxygen level having an oxygen concentration of less than about 1 mole% (mol%); less than about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol%; less than about 0.001 mol%; or less than about 0.0000001 mol%.

Figure 1D shows an illustrative alternative conduit 1030 that includes a coated particle classifier 1090, according to one implementation. The coated particle classifier 1090 can include any number of devices,

systems, or combinations of systems and devices suitable for separating, classifying, sorting, or otherwise apportioning the separated coated particles 1032. Such classification may be based at least in part on one or more defined physical properties of the separated coated particles 1032, one or more defined compositional properties of the separated coated particles 1032, or any combination thereof. For example, the coated particle classifier 1090 may apportion the separated coated particles 1032 into a defined number of fractions based on the respective diameters of the separated coated particles 1032.

At times, the environment in the coated particle classifier 1090 can be maintained at a low oxygen level having an oxygen concentration of less than 20 volume percent (vol%). At other times, the environment in the coated particle classifier 1090 can be maintained at a very low oxygen level having an oxygen concentration of less than about 1 mole% (mol%); less than about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol%; less than about 0.001 mol%; or less than about 0.0000001 mol%.

At times, within the coated particle classifier 1090, all or a portion of the separated coated particles 1032 are apportioned into at least the first portion of separated coated particles 1034 for subsequent transfer to the coated particle melter 1050 and the second portion of separated coated particles 1038, at least a portion of which are subsequently recycled to the particulate bed in the reactor 30.

Figure 1E shows an illustrative alternative conduit 1030 that includes a coated particle accumulator 1080 and a coated particle classifier 1090, according to one implementation. All or a portion of the separated coated particles 1032 are directed to the coated particle accumulator 1080. The separated coated particles 1032 are transferred from the coated particle accumulator 1080 to the coated particle classifier 1090 on demand, intermittently, periodically, or continuously.

At times, the environment in the coated particle accumulator 1080 and the coated particle classifier 1090 can be maintained at a low oxygen level having an oxygen concentration of less than 20 volume percent (vol%). At other times, the environment in the coated particle accumulator 1080 and the coated particle classifier 1090 can be maintained at a very low oxygen level having an oxygen concentration of less than about 1 mole% (mol%); less than about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol%; less than about 0.001 mol%; or less than about 0.0000001 mol%.

Figure 1F shows an illustrative alternative conduit 1030 that includes a coated particle classifier 1090 and a coated particle grinder 1096, according to one implementation. The coated particle classifier 1090 can apportion the separated coated particles 1032 into the first portion of separated coated particles 1034 that is subsequently transferred to the coated particle melter 1050 and the second portion of separated coated particles 1038. In at least some implementations, at least some of the second portion of separated coated particles 1038 may include coated particles for recycle to the particulate bed 1004 in reactor 1002 or the mechanically fluidized particulate bed 20 in the mechanically fluidized reactor 30.

However, at times, the diameter of at least some of the second portion of separated coated particles 1038 may be too large for recycle to the particulate bed 1004 or the mechanically fluidized particulate bed 20. In such instances, the coated particle classifier 1090 may further classify the second portion of separated coated particles 1038 into either a first fraction 1092 if the coated particle diameter exceeds a defined threshold (i.e., a large diameter fraction) or a second fraction 1094 if the coated particle diameter is less than the defined threshold (i.e., a small diameter fraction). All or a portion of the first fraction 1092 can be transferred to the coated particle grinder 1096 where the diameter of the coated particles is reduced to a size suitable for recycle to the particulate bed 1004 in reactor 1002 or to the mechanically fluidized particulate

bed 20 in the mechanically fluidized reactor 30. In such instances, all or a portion of the reduced diameter coated particles discharged by the coated particle grinder 1096 can be combined with all or a portion of the second (small diameter) fraction 1094 for recycle to the particulate bed 1004 in reactor 1002 or to the mechanically fluidized particulate bed 20 in the mechanically fluidized reactor 30. Alternatively or additionally, all of the second portion of separated coated particles 1038 is transferred to the coated particle grinder 1096.

All or a portion of the second portion of coated particles 1038 may be transferred to from the coated particle classifier 1090 to the coated particle grinder 1096 on demand, intermittently, periodically, or continuously. All or a portion of the first portion of separated coated particles 1034 may be transferred from the coated particle classifier 1090 to the coated particle melter 1050 on demand, intermittently, periodically, or continuously. All or a portion of the separated coated particles 1032 may be directed to the coated particle classifier 1090 on demand, intermittently, periodically, or continuously.

At times, the environment in the coated particle classifier 1090 and the coated particle grinder 1096 can be maintained at a low oxygen level having an oxygen concentration of less than 20 volume percent (vol%). At other times, the environment in the coated particle classifier 1090 and the coated particle grinder 1096 can be maintained at a very low oxygen level having an oxygen concentration of less than about 1 mole% (mol%); less than about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol%; less than about 0.001 mol%; or less than about 0.0000001 mol%.

Figure 1G shows an illustrative alternative conduit 1030 that includes the coated particle accumulator 1080, the coated particle classifier 1090, and the coated particle grinder 1096 according to another implementation. Additionally or alternatively to any one or more of the explanations provided above with regard to any one or more of Figures 1E and 1F, all or a portion of the separated coated particles 1032 may be directed to

the coated particle accumulator 1080. The separated coated particles 1032 may be transferred from the coated particle accumulator 1080 to the coated particle classifier 1090 on demand, intermittently, periodically, or continuously.

At times, the environment in any one or more of the coated particle accumulator 1080, the coated particle classifier 1090, or the coated particle grinder 1096 can be maintained at a low oxygen level having an oxygen concentration of less than 20 volume percent (vol%). At other times, the environment in any one or more of the coated particle accumulator 1080, the coated particle classifier 1090, or the coated particle grinder 1096 can be maintained at a very low oxygen level having an oxygen concentration of less than about 1 mole% (mol%); less than about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol%; less than about 0.001 mol%; or less than about 0.0000001 mol%.

Returning now to Figure 1A, the production and/or transport of coated particles 22 and/or the separated coated particles 1032 in low oxygen content environments such as the reactor 1002 and/or the conduit 1030 significantly reduces the likelihood of oxide formation and/or contaminant deposition, adhesion, and/or adsorption on the surfaces of the coated particles 22 and/or the separated coated particles 1032, particularly smaller diameter coated particles. Advantageously, such permits transferring coated particles to the coated particle melter 1050, without removing such smaller diameter coated particles from the separated coated particles 1032, while minimizing or avoiding the issues associated with the (relatively) greater quantity of oxides (and their inherent melt problems discussed above) that would otherwise be introduced to the coated particle melter 1050. Because the formation of an oxide layer on the separated coated particles 1032 is minimized or even eliminated, isolation of small diameter coated particles is not necessarily required and small diameter separated coated particles can, contingent upon solving dusting problems and melting problems due to low particle density, be charged to the

coated particle melter 1050 without having an adverse effect on final crystal quality and/or composition.

For example, in some crystal production methods, separated coated particles having diameters of about 400 μm to about 4000 μm may be deemed “desirable” while coated particles having a diameter of about 400 μm or less are deemed “dust” and undesirable within coated particle melters. The smaller diameter separated coated particles are problematic for several reasons, including the difficulty of melting the smaller diameter coated particles and/or the increase in thermal energy input required to melt smaller diameter separated coated particles that float within coated particle melters. It is theorized that smaller diameter particles including an oxide layer require additional thermal energy input due to the elevated melting point due to the presence of the oxide layer.

In another example, small diameter coated particles produced in a hydraulically fluidized bed reactor may have a greater tendency to detrimentally suspend within the environment in coated particle melters than smaller diameter separated coated particles produced in the mechanically fluidized bed reactor. The tendency for smaller diameter coated particles produced in a hydraulically fluidized bed reactor to suspend within coated particle melters may be attributable, at least in part, to the relatively low bulk density of smaller diameter coated particles produced in a hydraulically fluidized bed reactor.

The systems and methods described herein advantageously produce the coated particles 22 and maintain the separated coated particles 1032 in environments having low oxygen levels or very low oxygen levels. Further, the ability to charge smaller diameter separated coated particles produced in the mechanically fluidized bed reactors described herein directly to the coated particle melter 1050 minimizes, reduces, or even eliminates classification and removal of smaller diameter coated particles from the coated particle melter charge. Because classification of coated particles (e.g., coated particles produced in a hydraulically fluidized bed) introduces contaminants

such as metal atoms and ions to coated particles, the ability to charge smaller diameter separated coated particles (e.g., coated particles 22 produced in the mechanically fluidized particulate bed 1004) without classification reduces the contaminants carried by the first portion of separated coated particles 1034 into
5 the coated particle melter 1050 making possible the production of second chemical species crystals having low contaminant levels or very low contaminant levels.

Such coated particles 22 and separated coated particles 1032, having minimal or no oxide layer and minimal or no contaminants due to
10 surface contact or exposure in the conduit 1030, permit the rapid melting of smaller particles in the coated particle melter 1050, thereby enabling the use of even small diameter particles in the crystal production process without the attendant melting and contamination issues associated with coated particles having an oxide layer and surface contact or exposure in the conduit 1030. It is
15 possible that, at times, the improved “meltability” or improved melt characteristics of such smaller diameter coated particles and such smaller diameter separated coated particles produced in the mechanically fluidized particulate bed reactor is at least partially attributable to the higher density of the coated particles 22 and separated coated particles 1032, including the small
20 diameter coated particles and small diameter separated coated particles, and the consequent reduced propensity to form dust in the coated particle melter 1050. It is possible that, at times, the “meltability” of such smaller diameter coated particles and such smaller diameter separated coated particles produced in the mechanically fluidized particulate bed reactor 30 is attributable
25 at least in part to the morphology of the coated particles 22 produced in the mechanically fluidized particulate bed 1004.

Additionally, the crystal production system 1000 preferentially generates larger diameter separated coated particles which have a much lower surface area/mass ratio than comparatively smaller diameter separated coated
30 particles. Consequently, even if an oxide layer forms on the separated coated

particles 1032, the effects of such an oxide layer in the coated particle melter 1050 are advantageously mitigated by the significantly greater mass of second chemical species carried by each separated coated particle included in the first portion of separated coated particles 1034.

5 One or more exhaust gas systems 1010 removes as an exhaust gas at least a portion of any accumulated gases from the upper chamber 1003 of the reactor 1002. Such accumulated gases can include, but are not limited to, unconverted first gaseous chemical species, one or more diluents, and/or one or more third gaseous chemical species byproducts resulting from the
10 conversion of the first gaseous chemical species to the second chemical species. The one or more exhaust gas systems 1010 may include one or more gas separators (e.g., selectively permeable membranes, filters, and the like) that selectively separate all or a portion of the unconverted first gaseous chemical species, one or more diluent(s), and/or one or more gaseous
15 byproducts from the exhaust gas. All or a portion of the separated gaseous byproducts may be recycled, for example as one or more diluents added to the first gaseous chemical species.

In addition, the exhaust gas removed from the upper chamber 1003 may include particulate matter, for example particulates from the
20 particulate bed 1004. The exhaust gas system 1010 may include one or more solids separators (e.g., cyclonic separators, baghouses, and the like) to remove such particulates and/or particles entrained in the exhaust gas. At times, all or a portion of the removed particles or particulates may be recycled to the particulate bed 1004.

25 The conduit 1030 can include one or more devices, systems, or combinations of systems and devices suitable for at least transferring at least a portion of the separated coated particles 1032 to the coated particle melter 1050 while maintaining the separated coated particles 1032 in an environment having a low oxygen level or very low oxygen level and, attributable at least in
30 part to the elimination of a classification system, process, or device, a low

contaminant level or very low contaminant level. At times, the conduit 1030 may include additional devices, systems, or combinations of systems and devices for accumulation and/or storage of separated coated particles 1032, classification of separated coated particles 1032, and/or physical size reduction of separated coated particles 1032. The conduit 1030 may be a lined vessel, container, carboy, sack, bag, jug, or similar capable of maintaining the separated coated particles 1032 in the environment having a low oxygen level or very low oxygen level and a low contaminant level or very low contaminant level. At times, the conduit 1030 may be lined. Such liners may include, but are not limited to: silicon, quartz, graphite, silicon nitride, silicon carbide, molybdenum disilicide, polyethylene, or similar.

The conduit 1030 can include a housing 1040 having an interior space 1042 defining an environment in which the separated coated particles 1032 at least temporarily reside. At times, the environment in the interior space 1042 is maintained at a low oxygen level having an oxygen concentration of less than 20 volume percent (vol%) oxygen. At other times, the environment in the interior space 1042 is maintained at a very low oxygen level environment having an oxygen concentration of less than about 1 mole% (mol%); less than about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol%; less than about 0.001 mol%; or less than about 0.0000001 mol%.

At times, the conduit 1030 is simultaneously hermetically sealed to the reactor 1002 and to the coated particle melter 1050. Figures 1A-1G depict such an installation where the coated particle collection system 130 provides at least a portion of the conduit 1030 and is shown hermetically sealed to the reactor via the coated particle removal tube 132.

Alternatively, the conduit 1030 can include a moveable or transportable housing (not shown) that is hermetically sealable to the reactor 1002 to receive coated particles 22 from the particulate bed 1004. The transportable housing is moved proximate and hermetically sealed to the

coated particle melter 1050 to discharge the first portion of separated coated particles 1034 to the coated particle melter 1050.

The coated particle melter 1050 heats the first portion of separated coated particles 1034 received from the conduit 1030 to a temperature equal to or in excess of the melting temperature of the second chemical species. The coated particle melter 1050 includes a housing 1052 defining an interior space 1054. At times, the melter can include one or more thermal energy emitting devices that are used to heat the first portion of separated coated particles 1034 to a temperature equal to or in excess of the melting temperature of the second chemical species deposited on coated particles of the first portion of separated coated particles 1034. At other times, the coated particle melter 1050 can include one or more inductive, radio frequency, microwave, or other electromagnetic energy emitting or producing devices suitable for increasing the temperature of the first portion of the separated coated particles 1034 to a temperature equal to or in excess of the melting temperature of the second chemical species.

In some instances, for example a Czochralski (CZ) crystal production process, a lined quartz crucible can receive the first portion of separated coated particles 1034. In such instances, the quartz crucible can include one or more linings or similar coatings (e.g., a barium doped quartz or silicon nitride coating) that advantageously mitigate the dissolution of silicon dioxide from the crucible to the molten second chemical species pool 1060.

At times, within the coated particle melter 1050, dissolved silicon dioxide (*e.g.*, silicon dioxide dissolved from a quartz crucible or carried into the melt with the first portion of separated coated particles 1034) is converted to silicon monoxide, which at typical melt temperatures is in a gaseous state. The silicon oxide tends to migrate toward the surface of the molten second chemical species pool 1060, and is substantially swept and removed from the melt pool 1060 by an inert gas sweep. Oxygen that is not removed from the molten second chemical species pool 1060 can incorporate into second chemical

species crystal boule as the puller 1072 pulls the second chemical species crystal boule from the molten second chemical species pool 1060. Oxygen introduced as a layer of silicon dioxide present on at least some of the first portion of separated coated particles 1034 can significantly add to the oxygen
5 from the crucible 428, and significantly increase potential for oxygen contamination in the second chemical species silicon crystal boule with concomitant adverse effect on the quality of the second chemical species crystal. This oxygen contamination may render all or a portion of the second chemical species crystal unsuitable for use in semiconductor or solar cell
10 fabrication.

Surface contaminants on some or all of the first portion of separated coated particles 1034, including metal atoms and/or ions, do not volatilize out of the molten second chemical species but instead concentrate in the molten second chemical species present in the crucible 428.
15 Contaminants, including surface contaminants carried in by coated particles, reaching a defined threshold value at which crystal growth and/or quality is adversely impacted requires dumping the molten second chemical species. Accordingly, minimizing or eliminating the oxide layer and/or surface contaminants on the first portion of separated coated particles 1034 therefore
20 advantageously permits the extended, even continuous, use of the molten second chemical species pool 1060.

All or a portion of the exterior surfaces of the coated particle melter 1050 can include an insulative layer 1056. The coated particle melter 1050 and/or the crucible 428 may have one or more thermal or electromagnetic
25 energy emitting devices 1058 that can provide all or a portion of the energy used to increase the temperature and melt all or a portion of the first portion of separated coated particles 1034. In some instances, the melted coated particles form a molten second chemical species pool or reservoir 1060 in at least a portion of the coated particle melter 1050.

At times, the environment in the interior space 1054 of the coated particle melter 1050 is maintained at a low oxygen level in which the oxygen concentration is less than 20 volume percent (vol%). At other times, the environment in the interior space 1054 of the coated particle melter 1050 is maintained at a very low oxygen level in which the oxygen concentration is less than about 1 mole% (mol%); less than about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol%; or less than about 0.001 mol%.

In some instances, the crystal production device 1070 can be physically coupled and hermetically sealed to the coated particle melter 1050 and/or the crucible 428. For example, the crystal production device 1070 can, at times, include a crystal puller or similar device that uses the Czochralski method to form a second chemical species. The Czochralski method uses a second chemical species seed crystal that is inserted into the molten second chemical species pool 1060 and withdrawn at a controlled rate and, optionally, rotation such that a second chemical species ingot or boule forms (i.e., “grows”) on the seed crystal.

Using silicon as an illustrative example, controlling oxide levels and contamination levels in the molten silicon pool 1060 by limiting the formation of an oxide layer and/or the deposition of contaminants on the surfaces of the first portion of separated coated particles 1034 provides high quality crystal silicon boules with minimal contamination. When an oxide layer is present on the surface of the first portion of separated coated particles 1034 the level of oxygen contamination the product monocrystalline silicon boules can remain unacceptably high for an extended period of time (e.g., in excess of an hour) after start-up of the crystal production device 1070. The presence of contaminants, including but not limited to oxygen atoms, molecules containing oxygen, metal atoms, molecules containing metal atoms, carbon atoms and molecules containing carbon atoms, in a silicon boule compromise the quality of the boule and may render the silicon boule unsatisfactory for use in

semiconductor or solar cell fabrication. The systems and methods described herein advantageously minimize or even eliminate the presence of such contaminants in the silicon coated particles used in producing the silicon boules or monocrystalline silicon. Such highly pure coated particles, including coated
5 particles of smaller diameter that, if not for the systems and methods described herein, would otherwise need to be excluded from the monocrystalline silicon production process, may be beneficially used in the production of monocrystalline silicon using any current or future developed crystal growing or production process.

10 For example, it is estimated that the level of oxide contamination attributable to the oxide layer on the first portion of separated coated particles 1034 introduced to the coated particle melter 1050 can be five times the level of oxide contamination attributable to other sources such as dissolution from the crucible 428 in which the first portion of separated coated particles 1034 are
15 melted. This problem is even more pronounced in continuous process schemes where granules are batch-wise or continuously recharged to the coated particle melter 1050. At other times, the crystal production device 1070 can use the Bridgman–Stockbarger crystal growing method in which a second chemical species seed crystal is introduced to a crucible 428 containing a reservoir or
20 pool of the molten second chemical species and the reservoir or pool is cooled at a defined rate to crystallize the second chemical species. Such a crystal grower may be particularly advantageous for growing doped second chemical species crystals, for example gallium arsenide doped silicon crystals.

Figure 2 shows an illustrative crystal production system 1100 that
25 includes a mechanically vibrated bed reactor 300 (e.g., mechanically vibrated fluidized bed reactor, mechanically vibrated packed or compressed bed reactor) fluidly coupleable to a portable conduit 1130, according to an illustrated embodiment. The portable conduit 1130 is fluidly coupleable to the coated particle melter 1050 and therefore enables the production and transfer of
30 separated coated particles 1032 while maintaining the separated coated

particles 1032 in an environment having a low oxygen level or a very low oxygen level. Although a mechanically fluidized bed reactor 300 is illustrated with crystal production system 1100, any of the mechanically vibrated bed reactors described in detail in Figures 1–2 may be substituted. Also, while 5 illustrated in Figure 2 with serpentine baffles, the reactor may employ radial baffles or may omit baffles.

The control system 190 may be communicably and operably coupled to the mechanically fluidized bed reactor 300, the coated particle melter 1050, and the crystal production device 1070. The control system 190 10 coordinates the operation of the mechanically fluidized bed reactor 300, the coated particle melter 1050, and the crystal production device 1070. For example, as the level in the crucible 428 containing the molten second chemical species pool 1060 decreases during crystal production, the control system 190 may cause the transfer of additional coated particles from the conduit 1130 to 15 the coated particle melter 1050 to maintain a defined minimum level in the crucible 428 containing the molten second chemical species pool 1060.

The control system 190 may alter, adjust, or control one or more process conditions in the mechanically fluidized bed reactor 300 to alter, adjust, or control the conversion of the first gaseous chemical species to the second 20 chemical species in the particulate bed 20. For example, the control system 190 may alter, adjust, or control one or more of: the temperature of the particulate bed 20, the temperature in the upper upper chamber 33 external to the particulate bed 20, the temperature in the lower upper chamber 34, a gas pressure (first gaseous chemical species, one or more optional diluent(s), 25 dopants, or combinations thereof) in the particulate bed 20, a flow rate of the first gaseous chemical species to the particulate bed 20, a ratio of the dopant to first gas, a flow rate of dopant, the temperature of the gas feed comprising first reactive species to the particulate bed 20, a ratio of the first gaseous chemical species to the one or more optional diluent(s) in the particulate bed 20.

The control system 190 may alter, adjust, or control the oscillatory frequency and/or the oscillatory displacement of the pan 12. Controlling the oscillatory frequency and/or displacement of the pan 12 enables the selective separation of coated particles 22 from the mechanically fluidized particulate bed 20 via the coated particle overflow conduit 132. For example, the control system 190 can alter, control, or adjust an oscillatory displacement and/or an oscillatory frequency along one or more of three orthogonal axes that define a three dimensional space. By varying the oscillatory displacement and/or frequency along two orthogonal axes, circular or elliptical oscillations are possible. By varying the oscillatory displacement and/or frequency along three orthogonal axes, helical, spiral, and similar are possible. At times, at least one of: a horizontal oscillatory displacement component or a vertical oscillatory displacement component to selectively separate coated particles 22 meeting one or more desired physical or compositional thresholds from the particulate bed 20. Such advantageously enables the selective retention of particulates and coated particles in the particulate bed 20 having a diameter of less than about 600 micrometers (μm); less than about 500 μm; less than about 400 μm; less than about 300 μm; less than about 200 μm; less than about 100 μm; less than about 50 μm; less than about 20 μm; less than about 10 μm; less than about 5 μm; or less than about 1 μm.

The control system 190 can alter, adjust or control the oscillatory frequency of the pan 12 to any frequency within a defined frequency range. For example, the control system 190 can alter, adjust or control the oscillatory frequency of the pan 12 to a defined frequency range that includes frequencies from about 1 cycle per minute to about 5000 cycles per minute. Suitable frequencies can, for example include: about 1 cycle per minute; about 5 cycles per minute; about 50 cycles per minute; about 100 cycles per minute; about 500 cycles per minute; about 1000 cycles per minute; or about 2000 cycles per minute to about 50 cycles per minute; about 100 cycles per minute; about 500 cycles per minute; about 1000 cycles per minute; about 2000 cycles per minute;

about 3000 cycles per minute; about 4000 cycles per minute; or about 5000 cycles per minute.

The control system 190 can alter, adjust, or control the oscillatory displacement of the pan 12 to have a horizontal component within a defined range. For example, the control system 190 can alter, adjust or control the horizontal oscillatory displacement of the pan 12 to a defined displace range that includes a horizontal displacement from about 0.01 inches to about 5 inches. Suitable amplitudes can, for example include, about 0.01 inches, about 0.03 inches; about 0.05 inches; about 0.1 inches; about 0.2 inches; about 0.3 inches; or about 0.5 inches to about 0.01 inches; about 0.05 inches; about 0.1 inches; about 0.3 inches; about 0.5 inches; about 0.9 inches; about 2 inches; or about 5 inches.

The control system 190 can alter, adjust, or control the oscillatory displacement of the pan 12 to have a vertical component within a defined range. For example, the control system 190 can alter, adjust or control the vertical oscillatory displacement of the pan 12 to a defined displace range that includes a vertical displacement from about 0.01 inches; about 0.03 inches; about 0.05 inches; about 0.1 inches; about 0.2 inches; about 0.3 inches; or about 0.5 inches to about 0.01 inches; about 0.05 inches; about 0.1 inches; about 0.3 inches; about 0.5 inches; about 0.9 inches; or about 2.0 inches.

The control system 190 can additionally alter or adjust the flow of a purge gas to the coated particle overflow conduit 132 to alter, adjust, or control the diameter of the coated particles 22 separated from the particulate bed 20. For example, the control system 190 may increase the flow of purge gas through the coated particle overflow conduit 132 into the particulate bed 20 to selectively increase the diameter of the coated particles 22 separated from the mechanically fluidized particulate bed 20. Conversely, the control system 190 may decrease the flow of purge gas through the coated particle overflow conduit 132 into the particulate bed 20 to selectively decrease the diameter of

the coated particles 22 separated from the mechanically fluidized particulate bed 20.

The crystal production system 1100 maintains an environment having a low oxygen level or a very low oxygen level and/or a low contaminant level or very low contaminant level in at least the upper chamber 33 of the mechanically fluidized bed reactor 30, the conduit 1130, and the coated particle melter 1050. In addition, one or more coatings, liners, or layers may be applied to all or a portion of the mechanically vibrated bed reactor 30, the conduit 1130, and the coated particle melter 1150/crystal production device 1170 to further minimize the migration of oxygen and other contaminants (e.g., metals) from the process equipment to the separated coated particles 1032 and/or the product crystalline second chemical species. The upper chamber 33 of the mechanically vibrated bed reactor 30, the conduit 1130, and the coated particle melter 1050 are maintained at low oxygen level relative to the ambient environment. Coated particles in the upper chamber 33 of the mechanically vibrated bed reactor 30, the conduit 1130, and the coated particle melter 1050 are maintained in an environment having a low oxygen level or a very low oxygen level. At times, the environment in the upper chamber 33 of the mechanically vibrated bed reactor 30, the conduit 1130, and the coated particle melter 1050 is maintained at a low oxygen level having an oxygen concentration of less than 20 volume percent (vol%). At other times, the environment in the upper chamber 33 of the mechanically vibrated bed reactor 30, the conduit 1130, and the coated particle melter 1050 is maintained at a very low oxygen level having an oxygen concentration of less than about 1 mole% (mol%); less than about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol% oxygen; less than about 0.001 mol%; or less than about 0.0000001 mol%.. Advantageously, by limiting the exposure of the coated particles 22 and the separated coated particles 1032 to oxygen, oxide formation on the external surfaces of the coated particles 22 and

the separated coated particles 1032 is beneficially minimized, reduced, or even eliminated.

Minimizing, limiting, or eliminating oxide formation on the external surfaces of the coated particles 22 and the separated coated particles 1032 is believed to beneficially improve the “meltability” of the coated particles by reducing the tendency of small separated coated particles to melt at elevated temperatures compared to pure silicon, and improves the quality of the molten second chemical species pool 1060 by reducing oxide contaminants.

Minimizing, limiting, or eliminating oxide formation on the external surfaces of the coated particles 22 and the separated coated particles 1032 beneficially eliminates the need to classify separated coated particles 1032 to limit the introduction of smaller diameter separated coated particles to the coated particle melter 1050 since the smaller particles will not have significant oxide buildup on their surfaces. Furthermore, the ability to selectively separate coated particles from the mechanically vibrated particulate bed 20 such that smaller diameter separated coated particles are retained in the mechanically vibrated particulate bed 20 provides a synergistic effect that further reduces or even eliminates the need to separate smaller diameter coated particles from the first portion of separated coated particles 1034 introduced to the coated particle melter 1050. By eliminating the need to classify separated coated particles, exposure to free oxygen during the classification process is eliminated, beneficially improving the quality of the resultant second chemical species crystals provided by the crystal puller 1072.

Figure 3 shows a high-level block flow diagram of an illustrative crystal production method 1200, according to one implementation. A particulate bed can include coated particles that include a non-volatile second chemical species formed by the thermal and/or chemical decomposition of a first gaseous chemical species in the particulate bed. The non-volatile second chemical species can include any number of elements or compounds, including but not limited to, germanium and germanium silicon mixtures in the form of

Si_xGe_y, silicon, polysilicon, silicon nitride, silicon carbide, or aluminum oxide (e.g., sapphire glass). At times, an oxide layer or oxide shell can form on some or all of the exposed surfaces of the coated particles upon exposure to a gas that includes free oxygen. For example, a silicon dioxide layer can form on
5 some or all exposed surfaces of polysilicon coated particles simply upon exposure to air. The presence of such oxide layers interferes with subsequent processing of the coated particles, such as melting silicon coated particles during the production of silicon boules. The crystal production method 1200 commences at 1202.

10 At 1204, coated particles 22 are separated from a heated particulate bed. In some instances, the coated particles 22 may be separated from a heated particulate bed 1004 in a upper chamber 1003 of a reactor 1002 such as that described in Figure 1A. In such instances, the coated particles 22 may be separated from the particulate bed 1004 using any current or future
15 developed separations technology. Such separations may be based in whole or in part on one or more physical properties of the coated particles 22, such as diameter, density, and the like. Such separations may be based in whole or in part on one or more compositional properties of the coated particles 22.

In other instances, the coated particles 22 may be separated from
20 a fluidized particulate bed 20 in a vibrated bed reactor 30. In such implementations, the fluidized bed reactor 30 can include a mechanically vibrated bed 20 disposed in the chamber 32, such as any of the mechanically vibrated bed reactors described in Figures 1–2. In the mechanically vibrated bed reactor 30, the coated particles 22 can be separated by adjusting one or
25 more parameters of the vibrated particulate bed 20. For example, the oscillatory frequency and/or the oscillatory displacement of a pan supporting the mechanically vibrated particulate bed 20 can be altered or adjusted to cause the separation of coated particles 22 having one or more desirable physical or compositional characteristics.

At 1206, the first portion of separated coated particles 1034 removed from the heated particulate bed 20 are conveyed to a coated particle melter 1050. In some implementations, the transfer of the first portion of separated coated particles 1034 is performed via a conduit 1030 that maintains the separated coated particles 1032 in an environment having either a low oxygen level or a very low oxygen level. At times, the environment in the conduit 1030 is at a low oxygen level having an oxygen concentration of less than 20 volume percent (vol%). At other times, the environment in the conduit 1030 is maintained at a very low oxygen level having an oxygen concentration of less than about 1 mole% (mol%); less than about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol%; less than about 0.001 mol%; or less than about 0.0000001 mol%.

Reducing the exposure of the separated coated particles 1032 to oxygen during the transport between the reactor and the coated particle melter 1050 beneficially reduces the formation of an oxide layer on the exposed surfaces of the separated coated particles 1032. Reducing or preventing the formation of an oxide layer on the separated coated particles 1032 provides numerous advantages that include: melting smaller diameter separated coated particles without the potential for a detrimental melting point temperature increase in the coated particle melter 1050; and reducing, or possibly eliminating, the need for classification of some or all of the separated coated particles 1032 prior to melting. The reduction or elimination of oxide formation and contamination of the separated coated particles 1032 improves the quality and/or consistency of crystals produced using the separated coated particles 1032 due to decreased oxide contaminants and reduced metal contaminants associated with classification systems, equipment, processes and/or methods. The crystal production method 1200 concludes at 1208.

Figure 4 shows an illustrated crystal production method 1300 in which a first gaseous chemical species is thermally decomposed in a vibrated particulate bed 20 that has been heated to a temperature in excess of a thermal

decomposition temperature of the first gaseous chemical species, according to an embodiment. The thermal decomposition temperature of the first gaseous chemical species is the temperature at which the first gaseous chemical species chemically decomposes to provide at least the second chemical
5 species. At times, the thermal decomposition of the first gaseous chemical species also produces one or more third gaseous chemical species reaction byproducts. The thermal decomposition of the first gaseous chemical species can be an endothermic process using thermal energy (i.e., heat) to break chemical bonds and thermally decompose the first gaseous chemical species
10 into a number of constituent components. The crystal production method 1300 commences at 1302.

At 1304, the particulate bed is vibrated to provide a vibrated particulate bed. At times, vibrated of the particulate bed can occur hydraulically via the passage of one or more fluids (i.e., one or more liquids or gases)
15 through the particulate bed at a flow rate (or superficial velocity) sufficient to vibrated the particulates present in the particulate bed. At other times, vibration of the particulate bed can occur mechanically by oscillating a pan 12 or other major horizontal surface 302 that carries the particulate bed at an oscillatory frequency and oscillatory displacement sufficient to impart fluid like properties
20 to the particulate bed to provide a mechanically vibrated particulate bed 20. When vibrated, the particulates in the vibrated particulate bed may demonstrate water like fluid properties such as flowability and circulation.

At 1306, one or more thermal energy emitting devices 14 increase the temperature of the vibrated particulate bed 20 above the thermal
25 decomposition temperature of the first gaseous chemical species. At times, the thermal energy emitting devices 14 may be positioned proximate a pan 12 or a major horizontal surface 302 carrying the vibrated particulate bed 20, in which case the one or more thermal energy emitting devices 14 indirectly heat the particulate bed by heating the pan or major horizontal surface. Such an
30 arrangement is particularly advantageous since the only reactor components

above the thermal decomposition temperature are proximate the vibrated particulate bed – where the thermal decomposition of the first gaseous chemical species is highly preferred. At times, the thermal energy emitting devices 14 may be positioned a distance from the fluidized particulate bed 20,
5 for example a convection or radiant heater.

At 1308, coated particles 22 are formed by thermally decomposing the first gaseous chemical species in the heated vibrated particulate bed 20. At times, the first gaseous chemical species is introduced directly to the heated vibrated particulate bed using a distribution header 350
10 and one or more injectors 356 positioned in the heated vibrated particulate bed 20. In some instances, the one or more injectors 356 can be insulated for example using a vacuum, insulative material, cooling fluid, or combinations thereof.

The first gaseous chemical species decomposes within the
15 heated vibrated particulate bed and deposits the nonvolatile second chemical species on the surfaces of the particulates, forming the plurality of coated particles 22 in the heated vibrated particulate bed. The coated particles 22 can then be selectively separated from the heated vibrated particulate bed and transferred to the conduit 1030. The crystal production method 1300 concludes
20 at 1310.

Figure 5 shows a high level block flow diagram of an illustrative crystal production method 1400 in which one or more optional diluents are provided to the heated vibrated particulate bed contemporaneous with the introduction of the first gaseous chemical species to the heated vibrated
25 particulate bed, according to an embodiment. At times, it is advantageous to provide minimum gas flow to the heated vibrated particulate bed, however feeding solely first gaseous chemical species may adversely impact the conversion to the second chemical species in the heated vibrated particulate bed. In such instances, one or more optional diluents may be used to provide
30 the desired gas flow through the heated vibrated particulate bed while

maintaining the conversion of the first gaseous chemical species to the second chemical species at desired levels. The crystal production method 1400 commences at 1402.

At 1404, one or more diluents are mixed with the first gaseous chemical species prior to thermally decomposing the first gaseous chemical species in the heated fluidized particulate bed. At times, the one or more diluents may be premixed with the first gaseous chemical species external to the heated vibrated particulate bed and introduced to the heated vibrated particulate bed via the number of injectors 356 as a mixture containing defined proportions of the one or more diluents and the first gaseous chemical species. At other times, the one or more diluents may be introduced to the heated vibrated particulate bed separate from the first gaseous chemical species. At such times, the circulation of the heated vibrated particulate bed can assist in mixing the one or more diluents and the first gaseous chemical species in the heated vibrated particulate bed.

The one or more diluents can include any chemically inert material that either does not impact the composition or physical characteristics of the second chemical species on the particles in the heated vibrated particulate bed or has an overall positive or desirable effect on the composition or physical characteristics of the second chemical species deposited on the particles in the heated vibrated particulate bed. At times, the one or more diluents may be chemically identical to one or more third gaseous chemical species byproducts. For example, hydrogen may be used as a diluent with a first gaseous chemical species gas such as silane. Silane generates hydrogen as a byproduct upon thermal decomposition in the heated vibrated particulate bed. Other inert gases suitable for use as a diluent include, but are not limited to nitrogen, helium, and argon. The crystal production method 1400 concludes at 1406.

Figure 6 shows a high level block flow diagram of an illustrative crystal production method 1500 in which one or more optional dopants are

provided to the heated vibrated particulate bed, according to an embodiment. At times, the one or more optional dopants may be added to the heated vibrated particulate bed contemporaneous with the introduction of the first gaseous chemical species to produce doped coated particles 22. At other times, the one or more optional dopants may be added to the heated vibrated particulate bed at times when the first gaseous chemical species is not added to produce doped coated particles 22. Dopants, particularly dopants used in the production of silicon crystals, produce desirable molecular flaws in the crystalline structure. Dopants include, but are not limited to boron, arsenic, phosphorus, and gallium. The crystal production method 1500 to produce doped coated particles commences at 1502.

At 1504, one or more dopants are mixed with the first gaseous chemical species in the heated vibrated particulate bed. At times, the one or more dopants may be premixed with the first gaseous chemical species external to the heated vibrated particulate bed and introduced to the heated vibrated particulate bed via a distribution header 350 and a number of injectors 356 as a mixture containing defined proportions of the one or more dopants and the first gaseous chemical species. At other times, the one or more dopants may be introduced to the heated vibrated particulate bed separate from the first gaseous chemical species. At such times, the one or more dopants and the first gaseous chemical species mix in the heated vibrated particulate bed. The crystal production method to produce doped coated particles 22 concludes at 1506.

Figure 7 shows a high level block flow diagram of an illustrative crystal production method 1600 in which a heated vibrated particulate bed is disposed in a chamber of a reactor vessel and the temperature in the chamber external to the heated vibrated particulate bed and the temperature of the first chemical species while external to the heated vibrated particulate bed are maintained at a temperature or temperatures that are lower than the thermal decomposition temperature of the first gaseous chemical species, according to

an embodiment. The production of coated particles 22 in the heated vibrated particulate bed takes advantage of the generation of the non-volatile second chemical species upon exposure of the first gaseous chemical species to a temperature greater than its thermal decomposition temperature. If other surfaces in the chamber housing the heated vibrated particulate bed are greater than the thermal decomposition temperature of the first gaseous chemical species, it is likely that second chemical species deposits will occur on those surfaces. Such deposits external to the heated vibrated particulate bed detrimentally impact yield and may compromise operating efficiency. The crystal production method 1600 commences at 1602.

At 1604, the heated vibrated particulate bed is disposed in a chamber 32 inside a reactor vessel 31. In some instances, the chamber 32 can be apportioned into multiple chambers, for example an upper chamber 33 and a lower chamber 34 created by apportioning the chamber 32 using a flexible member 42 (e.g., flexible metal bellows). In other instances, the chamber 32 may include a unitary (i.e., undivided) chamber inside the reactor vessel 31. At times, the pan 12 or major horizontal surface 302 supporting the heated vibrated particulate bed inside the chamber 32 is operably coupled to a transmission 50 that is used to oscillate the pan 12 or major horizontal surface 302 at one or more defined oscillatory frequencies or oscillatory displacements.

At 1606, the chamber 32 external to the heated vibrated particulate bed is maintained at a temperature less than the thermal decomposition temperature of the first gaseous chemical species. At times, the temperature of the chamber 32 may be maintained below the thermal decomposition temperature of the first gaseous chemical species via one or more active thermal energy transfer devices (e.g., cooling coils, cooling jackets, and the like), one or more passive thermal energy transfer devices (e.g., extended surface cooling fins and the like), or combinations thereof. At times, the control system 190 may alter or adjust the temperature of the chamber 32 external to the heated vibrated particulate bed using one or more active thermal

energy transfer devices. The crystal production method 1600 concludes at 1608.

Figure 8 shows a high level block flow diagram of an illustrative crystal production method 1700 in which a second portion of the separated
5 coated particles 1038 are recycled, as seed particulate, to the heated particulate bed 1004, according to an embodiment. The physical and/or compositional properties of the separated coated particles 1032 can form a distribution (e.g., a Gaussian distribution) about a mean or median value. For example, the separated coated particles 1032 can have a variety of diameters
10 that form a Gaussian distribution about a mean diameter. At times, it may be preferable to forward a first portion of the separated coated particles 1034, for example those having a diameter greater than a defined threshold, to the coated particle melter 1050. At such times, it may be preferable to recycle a second portion of the separated coated particles 1038, for example those
15 having a diameter less than a defined threshold, back to the heated particulate bed 1004. The small diameter coated particles included in the second portion of the separated coated particles 1038 may function as seed particles for the deposition of additional layers of the second chemical species in the heated particulate bed. The crystal production method 1700 commences at 1702.
20 At 1704, the separated coated particles 1032 are classified, apportioned, sorted, separated or segregated into at least a first portion of separated coated particles 1034 and a second portion of separated coated particles 1038. Such separation or segregation may, at times, occur at least partially within the reactor, the conduit 1030, or any combination thereof. The
25 classification of the separated coated particles 1032 into the first portion of coated separated particles 1034 and the second portion of separated coated particles 1038 can occur in an environment having a low oxygen level or a very low oxygen level, thereby reducing or even eliminating the formation of an oxide layer or “shell” on the exposed surfaces of the separated coated particles 1032.
30 At times, the classification of the separated coated particles 1032 is performed

in a low oxygen level environment having an oxygen concentration of less than 20 volume percent (vol%). At other times, the classification of the separated coated particles 1032 is performed in a very low oxygen level environment having an oxygen concentration of less than about 1 mole% (mol%); less than 5 about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol%; less than about 0.001 mol%; or less than about 0.0000001 mol%.

At times, the second portion of separated coated particles 1038 may contain coated particles having diameters too large for use as seed 10 particulates in the heated particulate bed. At such times, some or all of the second portion of separated coated particles 1038 may be further divided into a first (i.e., large diameter) fraction of coated particles 1092 that are subsequently subjected to a size reduction process, for example using a coated particle grinder 1096 prior to recycle to the heated particulate bed, and a second (i.e., 15 smaller diameter) fraction of coated particles 1094 that are recycled directly to the heated particulate bed. The crystal production method 1700 concludes at 1706.

Figure 9 shows a high level block flow diagram of an illustrative crystal production method 1800 in which the first portion of separated coated 20 particles 1034 is melted in the coated particle melter 1050 and one or more second chemical species crystals are formed using the melted second chemical species, according to an embodiment. The chemical vapor deposition of second chemical species on the particulates in the heated particulate bed creates a substantially pure layer of second chemical species on each of the 25 separated coated particles 1032. The substantially oxygen and contaminant free separated particles 1032 made possible by handling the separated coated particles 1032 in an environment maintained at a low oxygen level or a very low oxygen level and a low contaminant level or very low contaminant level beneficially provides the ability to grow high purity second chemical species 30 crystals in the crystal production device 1070. Advantageously, the high purity

separated coated particles 1032 are amenable to use in many different crystal production devices or processes, including, but not limited to the Czochralski crystal production process, the Float Zone ("FZ") crystal production process, and directional crystal solidification processes such as the Bridgman-
5 Stockbarger production process. The crystal production method 1800 commences at 1802.

At 1804, the conduit 1030 deposits in or otherwise transfers to the coated particle melter 1050 and/or the crystal production device 1070 at least a portion of the first portion of separated coated particles 1034. The conduit 1030
10 is hermetically sealed to the coated particle melter 1050 and/or crystal production device 1070, thus the transfer of the first portion of coated particles 1038 is performed in an environment having a low oxygen level or a very low oxygen level. At times, the environment in the conduit 1030 is maintained at a low oxygen level having an oxygen concentration of less than 20 volume
15 percent (vol%). At other times the environment in the conduit 1030 is maintained at a very low oxygen level having an oxygen concentration of less than about 1 mole% (mol%); less than about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol%; less than about 0.001 mol%; or less than about 0.0000001 mol%. Handling the first portion of
20 the separated coated particles 1034 in an environment maintained at a low oxygen level or a very low oxygen level beneficially reduces or eliminates the formation of an oxide layer on the external surfaces of the first portion of separated coated particles 1034.

In some implementations, the coated particle melter 1050 heats
25 the first portion of separated coated particles 1034 above the melting temperature of the second chemical species. In such implementations, the melted coated particles form a molten second chemical species pool 1060 in the crucible 428 of the coated particle melter 1050. In such implementations, the molten second chemical species pool 1060 may be maintained in a reduced
30 free oxygen environment and a reduced contaminant environment in the coated

particle melter 1050 to reduce the undesirable formation of oxides. At times, given the relatively high purity of the molten second chemical species pool 1060, one or more dopants may be added to the molten second chemical species pool 1060 in the coated particle melter 1050 and/or crystal production device 1070.

At 1806 the crucible 428 containing the molten second chemical species pool 1060 is used to produce or grow one or more second chemical species crystals. At times, the one or more second chemical species crystals are substantially pure, crystalline second chemical species which may or may not contain one or more dopants dependent upon whether dopants were introduced to the molten second chemical species pool 1060. At times, a puller 1072 draws, pulls, or otherwise forms the one or more second chemical species crystals from the molten second chemical species pool 1060 using any current or future crystal production process, for example the Czochralski process in which crystals are drawn from the molten second chemical species pool 1060. At other times, one or more second chemical species crystals may be formed using the first portion of separated coated particles 1034 in one or more directional solidification crystallization processes such as the Bridgman-Stockbarger or Float Zone processes in which the molten second chemical species reservoir is cooled at a defined rate and in a defined directional pattern to create the crystalline second chemical species. The crystal production method 1800 concludes at 1808.

Figure 10 shows a high level block flow diagram of an illustrative crystal production method 1900 in which at least a portion of the first gaseous chemical species added to the heated particulate bed spontaneously self nucleates, propagating the particulate bed, and reducing or even eliminating the need for seed particulate addition to the heated particulate bed, according to an embodiment. Typically, fluidized beds require the addition of seed particulates to replace particulates lost through production (*i.e.*, removed from the bed as

coated particles) and particulates that escape the bed (e.g., particulates that become entrained in a fluid passed through the bed).

A mechanically fluidized particulate bed advantageously offers significantly lower superficial gas velocities than a comparable hydraulically fluidized particulate bed because the gas (i.e., the first gaseous chemical species with or without diluent) in the mechanically fluidized particulate bed is not relied upon to fluidize the bed. Consequently, smaller diameter particulates are advantageously retained in the mechanically fluidized particulate bed and are able to serve as seed particles for the deposition of the second chemical species. In fact, process conditions in the mechanically vibrated fluidized or packed particulate bed 20 may be adjusted to preferentially cause the spontaneous self-nucleation of at least a portion of the first gaseous chemical species introduced to the particulate bed, thereby reducing or even eliminating the need for seed particulate addition to the mechanically fluidized particulate bed. The crystal production method 1900 commences at 1902.

At 1904, one or more process conditions within a mechanically vibrated particulate bed 20 are adjusted, altered, or controlled to advantageously and preferentially cause the spontaneous self-nucleation second chemical species seed particulates using the first gaseous chemical species introduced to the mechanically vibrated fluidized or packed particulate bed 20. Such process conditions may include the pressure and/or temperature maintained in the mechanically vibrated fluidized or packed particulate bed 20. Such process conditions may include the feed rate of the first gaseous species. Such process conditions may include the oscillatory frequency and/or oscillatory displacement of the mechanically vibrated fluidized or packed particulate bed 20. Such process conditions may include a ratio of the first gaseous chemical species to one or more diluents added to the mechanically vibrated fluidized or packed particulate bed 20.

The spontaneous formation of self-nucleated seed particulates in the mechanically vibrated fluidized or packed particulate bed 20

advantageously reduces or even eliminates the need for the external addition of seed particulates to the mechanically vibrated fluidized particulate or packed bed 20. Eliminating the need for the external addition of seed particulates advantageously permits the operation of the mechanically vibrated fluidized or
5 packed particulate bed 20 in a closed, reduced free oxygen, environment. The ability to operate the vibrated bed in a closed environment advantageously makes possible the production of high purity coated particles and also makes possible the addition of one or more dopants to the mechanically vibrated fluidized or packed particulate bed 20 to produce doped coated particles in the
10 mechanically vibrated fluidized or packed particulate bed 20 — both of which offer significant advantages over conventional hydraulic fluidized bed production methods. The crystal production method 1900 concludes at 1906.

Figure 11 shows a high level block flow diagram of an illustrative crystal production method 2000 in which a mechanically vibrated fluidized or
15 packed particulate bed 20 generates second chemical species coated particles which are separated from the mechanically vibrated fluidized or packed particulate bed 20 and conveyed to a melter without exposing the coated particles to atmospheric oxygen, according to an embodiment. The crystal production method 2000 commences at 2002.

20 At 2004, an oscillatory frequency and/or an oscillatory displacement of a retention volume containing a mechanically vibrated fluidized or packed particulate bed 20 are adjusted to maintain the mechanically fluidized particulate bed 20 and also to separate coated particles 22 having one or more desirable or preferable physical and/or compositional characteristics from the
25 mechanically vibrated fluidized or packed particulate bed 20. For example, the oscillatory displacement of the retention volume may be adjusted along a single component axis of motion (e.g., along a horizontal component axis of displacement or along a vertical component axis of displacement) or along two or more component axes of motion (e.g., along a horizontal component axis of
30 displacement and along a vertical component axis of displacement). In another

example the oscillatory frequency of the retainment volume may be adjusted either upwards or downwards to achieve a desired coated particle separation.

At 2006, coated particles 22 are separated from the mechanically vibrated fluidized or packed particulate bed 20. At times, such separation may be achieved by overflowing at least a portion of the coated particles 22 into one or more hollow coated particle overflow tubes 132, each having at least one respective inlet positioned in the retainment volume. At other times, such separation may be achieved by overflowing at least a portion of the coated particles 22 over a perimeter wall or weir of the retainment volume (e.g., a peripheral wall or weir of a pan that forms at least a portion of the retainment volume). At times, the separated coated particles 1032 are collected in the conduit 1030 for transport to the coated particle melter 1050. At other times, as depicted in Figure 1B, a coated particle melter 1050 is hermetically sealed to the reactor 30 such that the coated particle melter 1050 directly receives the separated coated particles 1032.

At 2008, the conduit 1030 moves or otherwise transports, in a reduced free oxygen environment, at least a first portion of separated coated particles 1034 to the coated particle melter 1050. At times, the environment in the conduit 1030 is maintained at a low oxygen level having an oxygen concentration of less than 20 volume percent (vol%). At other times the environment in the conduit 1030 is maintained at a very low oxygen level having an oxygen concentration of less than about 1 mole% (mol%); less than about 0.5 mol%; less than about 0.3 mol%; less than about 0.1 mol%; less than about 0.01 mol%; less than about 0.001 mol%; or less than about 0.0000001 mol%. Handling the first portion of separated coated particles 1034 in an environment maintained at a low oxygen level or a very low oxygen level beneficially reduces or eliminates the formation of an oxide layer on the external surfaces of the first portion of separated coated particles 1034. The crystal production method 2000 concludes at 2010.

The systems and processes disclosed and discussed herein for the production of silicon have marked advantages over systems and processes currently employed. The systems and processes are suitable for the production of either semiconductor grade or solar grade silicon. The use of high purity silane as the first chemical species in the production process allows a high purity silicon to be produced more readily. The system advantageously maintains the silane at a temperature below the thermal decomposition temperature (e.g., 400ºC, 450ºC, or higher) until the silane enters the mechanically vibrated particulate bed. By maintaining process and equipment surface temperatures outside of the mechanically vibrated particulate bed below the thermal decomposition temperature of silane, the overall conversion of silane to usable polysilicon deposited on the particles within the mechanically vibrated particulate bed is increased, and parasitic conversion losses and operational problems attributable to decomposition of silane and deposition of polysilicon on other surfaces within the reactor are minimized.

The mechanically vibrated bed systems and methods described herein greatly reduce or eliminate the formation of ultra-fine poly-powder (e.g., 0.1 micron in size) external to the mechanically vibrated particulate bed 20 since the temperature of the gas containing the first chemical species is maintained below the auto-decomposition temperature of the first chemical species until injected into the mechanically vibrated bed. Additionally, the temperature within the chamber 32 is also maintained below the thermal decomposition temperature of the first chemical species further reducing the likelihood of auto-decomposition. Silane also provides advantages over dichlorosilane, trichlorosilane, and tetrachlorosilane for use in making high purity polysilicon. Silane is much easier to purify and has fewer contaminants than dichlorosilane, trichlorosilane, or tetrachlorosilane. Because of the relatively low boiling point of silane, it can be readily purified which reduces the tendency to entrain contaminants during the purification process as occurs in the preparation and purification of dichlorosilane, trichlorosilane, or tetrachlorosilane. Further, certain processes for the production of

trichlorosilane utilize carbon or graphite, which may carry along into the product or react with chlorosilanes to form carbon-containing compounds. Further, the silane-based decomposition process such as that described herein produces only a hydrogen by-product. The hydrogen byproduct may be directly recycled to the silane production process, reducing or eliminating the need for an off-gas treatment system. The elimination of off-gas treatment and the efficiencies of the mechanically vibrated bed process greatly reduce capital and operating cost to produce polysilicon. Savings of 40% in each are possible.

Pressure control for improved polysilicon melting

In one implementation of the dust suppression silicon production system and method of the present disclosure, the charging of the semiconductor grade-silicon granules is performed under high vacuum. Accordingly, such advantageously increases dust suppression compared to a charging phase under atmospheric conditions that is subsequently followed by a melting phase under vacuum (e.g., under 20 millibars (15 Torr) pressure).

Further, in one implementation of the dust suppression silicon production system and method of the present disclosure, an argon purge (or other inert gas purge) is not performed during the charging phase. Accordingly, such advantageously increases dust suppression compared to purging a melting chamber (e.g., melter 1050) during a charging phase. Alternatively, an inert gas purge may be continued or continued at a reduced rate under conditions where a vacuum is maintained during the charging phase, thereby improving dust suppression compared to a charging phase under atmospheric pressure while advantageously removing any SiO by-products formed by a reaction of silicon melt with a quartz crucible. In one implementation of the dust suppression silicon production system and method, the granules are under a high vacuum in the feed hopper (e.g., conduit 1030) as well as the melting chamber (e.g., melter 1050).

For example, a gas purging subsystem 416 may fluidly couple to the coated particle melter 1050 and/or the crucible 428. The gas purging

subsystem 416 may include, for example, one or more actuators 418 (*e.g.*, blower or mover). The actuator may fluidly couple to the coated particle melter 1050 and/or the crucible 428 via one or more gas headers 420.

One or more vacuum subsystems 422 may fluidly couple to the coated particle melter 1050 and/or the crucible 428. The vacuum subsystem 422 may include, for example, one or more vacuum motors 424. The vacuum motor 424 may pull fluid from the coated particle melter 1050 and/or the crucible 428 via one or more vacuum headers 426. The vacuum subsystem 426 may include one or more filters that further ensure clean exhaust from the coated particle melter 1050 and/or the crucible 428, thereby preventing potential health hazards in a surrounding work environment. The vacuum subsystem 422 may have a sufficient strength to maintain a high or very high vacuum in the coated particle melter 1050 and/or the crucible 428 during a gas purge by the gas purging subsystem 416.

A first valve 402 may selectively fluidly couple the reactor 1002 and the conduit 1030 via the hollow particle removal tube 132. In implementations involving the hollow particle recycle tube 406, a second valve 404 may selectively fluidly couple the reactor 1002 and the conduit 1030 via the hollow particle recycle tube 406. Additionally or alternatively third valve 408 may selectively fluidly couple the conduit 1030 and the coated particle melter 1050. Additionally or alternatively, the third valve 408 may selectively fluidly couple the conduit 1030 and the crucible 428.

Prior to and/or during the charging phase, the first valve 402 and/or the second valve 404 may close. Additionally or alternatively, the third valve 408 may open prior to and/or during the charging phase. The third valve 408 may open subsequent to or when the first valve 402 and/or the second valve 404 closes. The third valve 408 may move from a closed position where the third valve 408 hermetically seals the conduit 1030 from the coated particle melter 1050 and/or the crucible 428 to an open position where the third valve 408 fluidly couples the conduit 1030 to the coated particle melter 1050 and/or

the crucible 428, The third valve 408 may move from the closed position to the open position at a rate that causes a pressure in the conduit 1030 to gradually approach a pressure in the coated particle melter 1050 and/or the crucible 428. In particular, the third valve 408 may move at a rate that causes the gradual
5 approach to be smooth. Additionally or alternatively, the third valve 408 may move at a rate that causes the gradual approach to have an extended duration. The rate of such movement of the third valve 408 may be predetermined, e.g., via programming or hardware construction. As such, the third valve 408 may open slowly to equal a pressure between the conduit 1030 and the coated
10 particle melter 1050 and/or the crucible 428. Accordingly, the rate of such movement of the third valve 408 advantageously further suppresses dusting by preventing a sudden ingress of fluid and/or high rate of ingress of fluid from the conduit 1030 into the coated particle melter 1050 and/or the crucible 428. Additionally or alternatively to a predetermined rate of such movement of the
15 third valve 408, a sensor may measure the rate of ingress of fluid from the conduit 1030 into the coated particle melter 1050 and/or the crucible 428 while, responsive to the measured rate of ingress, the rate of such movement of the third valve 408 adjusts accordingly.

The conduit 1030 may include one or more conduit vacuum
20 subsystems that may create, decrease, maintain, substantially maintain, or increase a vacuum in the interior space 1042 of the conduit 68. Such conduit vacuum subsystem may include one or more elements, features, functions, or method acts that are similar to one or more elements, features, functions, or method acts that the present disclosure discloses with regard to the vacuum
25 subsystem 422. For example, prior to the third valve 408 opening, the conduit vacuum subsystem may equilibrate or substantially equilibrate one or more pressures or vacuums in the interior space 1042 of the conduit 1030 to such in the interior space 1054 of the melter 1050. Such reduces or eliminates fluid flow from or to the melter 1050, thereby advantageously reducing likelihood of

suspension of particles that one or more introduction tubes 1036 introduce to the interior space 1054 of the melter 1050.

One or more equilibrating tubes 409 may selectively fluidly couple the melter 1050 and the conduit 1030. The equilibrating tube 409 may connect
5 to such in one or more positions that precludes or substantially precludes particles from entering the equilibrating tube 409. Additionally or alternatively, one or more filters or screens may prevent passage of particles into the equilibrating tube 409. The equilibrating tube 409 may include one or more equilibrating valves 409a. After closure of one or more of the first valve 402 or
10 the second valve 404 (i.e., hermetically sealing the housing 1002 from the conduit 1030), the equilibrating valve 409a may open to fluidly couple the melter 1050 and the conduit 1030, and thereby equilibrate or substantially equilibrate one or more pressures or vacuums in the interior space 1042 of the conduit 1030 with the pressure or vacuum in the interior space 1054 of the
15 melter 1050. Such may occur prior to the opening of the third valve 408. Such may also occur subsequent to the conduit vacuum subsystem equilibrating or substantially equilibrating one or more pressures or vacuums in the interior space 1042 of the conduit 1030 to the pressure or vacuum in the interior space 1054 of the melter 1050. The equilibrating valve 409a may have one or more of
20 an opening rate or a rate control similar to the third valve 408. Such reduces or eliminates rate of flow fluid to the melter 1050 through one or more introduction tubes 1036, reducing or eliminating associated turbulence, and thereby advantageously reducing likelihood of suspension of particles that the introduction tube 1036 introduces to the interior space 1054. Responsive to
25 one or more of achieving equilibrium, achieving substantial equilibrium, opening of the third valve 408, or completion of the crucible charging phase or the crucible recharging phase, the equilibrating valve 409a may close.

At completion of the charging phase and/or subsequent to the completion of the charging phase, the third valve 408 may close. The first valve
30 402 and/or the second valve 404 may open subsequent to or when the third

valve 408 closes. The first valve 402 and/or the second valve 404 may move from a closed position where the first valve 402 and/or the second valve 404 hermetically seals the conduit 1030 from the reactor 1002 to an open position where the first valve 402 and/or the second valve 404 fluidly couples the conduit 1030 to the reactor 1002. The first valve 402 and/or the second valve 404 may move from the closed position to the open position at a rate that causes a pressure in the conduit 1030 to gradually approach a pressure in the reactor 1002. In particular, the first valve 402 and/or the second valve 404 may move at a rate that causes the gradual approach to be smooth. Additionally or alternatively, the first valve 402 and/or the second valve 404 may move at a rate that causes the gradual approach to have an extended duration. The rate of such movement of the first valve 402 and/or the second valve 404 may be determined, e.g., via programming or hardware construction. As such, the first valve 402 and/or the second valve 404 may open slowly to slowly equal a pressure between the conduit 1030 and the reactor 1002. Accordingly, the rate of such movement of the first valve 402 and/or the second valve 404 advantageously further suppresses turbulence in the reactor 1002 by preventing a sudden egress of fluid and/or high rate of egress of fluid from the reactor 1002 into the conduit 1030. Additionally or alternatively to a determined or limited rate of such movement of the first valve 402 and/or the second valve 404, a sensor may measure the rate of egress of fluid and/or high rate of egress of fluid from the reactor 1002 into the conduit 1030 while, responsive to the measured rate of egress, the rate of such movement of the first valve 402 and/or the second valve 404 adjusts accordingly. Some implementations may include one or more equalization lines with one or more associated valves that provide selective fluid communication to allow equalization of pressure between chambers and/or interior volumes of conduits. Such can be used to ensure a gradually adjustment of pressure, for example matching a pressure in the conduit to the high vacuum condition in the coated particle melter 1050 before or as part of charging of the crucible 428 in the coated particle melter 1050.

This can advantageously avoid loss of the high vacuum condition and/or further prevent or restrain dusting.

Additionally or alternatively, the one or more of the first valve 402, the second valve 404, or the third valve 408 may similarly operate with the
5 reactor 30.

In one implementation of the dust suppression silicon production system and method, while the feed hopper (e.g., conduit 1030) and the melting chamber (e.g., melter 1050) are both under high vacuum of 300 Torr or less, a valve is opened in a conduit that fluidly connects the feed hopper and a crucible
10 (e.g., crucible 428) inside the melting chamber. In another implementation of the dust suppression silicon production system and method, while the feed hopper and the internal melting chamber are both under high vacuum of 100 Torr or less, the valve is opened in the conduit that fluidly connects the feed hopper and the crucible inside the melting chamber. In still another
15 implementation of the dust suppression silicon production system and method, while the feed hopper and the internal melting chamber are both under high vacuum of 20 Torr or less, the valve is opened in the conduit that fluidly connects the feed hopper and the crucible inside the melting chamber. Optionally, and preferably, the pressure in the conduit (1030) and the melting
20 chamber (1050) is equilibrated before the valve is opened to make for a "bumpless" transfer of granules from the conduit into the melter.

The granules can, for example, flow due to gravity from the feed hopper (e.g., conduit 1030) into the crucible (e.g., crucible 428) within the melting chamber (e.g., melter 1050). Alternatively or additionally, one or more
25 active system or component may be employed, for example a vibrating table or belt, an auger, flaps, etc. Notably, dusting is suppressed during this process because fine material falls without lift in the absence of a gas. In one embodiment, fine material is defined as particles smaller than 500 microns. In another embodiment, fine material is defined as particles smaller than 300
30 microns. In still another embodiment, fine material is defined as particles

smaller than 100 microns. In yet another embodiment, fine material is defined as particles smaller than 20 microns.

By this method and system, the melter crucible 428 may be charged or recharged. When the charge or recharge is complete the valve may be closed. Alternately, in the case of continuous charging the valve is left open and a controlled feeding system, not shown, such as a rotating feeder valve controlled by the controller shown on Figure 2, continuously meters in an amount of granules necessary and sufficient to maintain the desired operating level of molten material in the melter (1050).

Technological improvements are produced by the unique vacuum implementation incorporated by the dust suppression silicon production system and method of the disclosed embodiments because the presence of a gas is required to create lift. Otherwise stated, because dusting is the uplift of fine particles due to drafts caused by convective gas currents, the specific implementation of a high vacuum during certain phases of the melting and charging processes results in the suppression of dusting. Because the silicon production industry evolved using open air polysilicon charging technology, the industry has not realized the technological improvements of using pressure control for improved silicon charging.

The techniques described herein apply to both doped or non-doped granule charging. Employing pre-doped charging where the particles are doped in the reactor before being introduced into the melter or puller may be particularly advantageous. For example, by pre-doping the particles or granules it is possible to achieve precise, accurate control of the level of dopant in the granules and hence in the crystal formed in the crystal production device (1070) Also for example, because the meter (1050) operates under a vacuum, dopant gases added to the melter (1050) would be removed from the melter system reducing efficient use of the dopant. As a further example, adding dopant gases to the reactor 1002 is possible and effective because the reactor is operated under atmospheric pressure or pressures significantly higher than atmospheric pressure; as a result, the retention time of the dopant gas is

sufficiently extended so as to allow for proper incorporation into the particles or granule.

For example, a doping subsystem 410 may supply dopant to the coated particle melter 1050 and/or the crucible 428. The dopant feed system 410 may include one or more dopant reservoirs 412 that store one or more of doped particles, non-doped particles, doped granules, non-doped granules, doped powder, non-doped powder, dopant gas, or non-doped gas for adjusting a level and/or mixture of dopant in the coated particle melter 1050 and/or the crucible 428. The dopant reservoir 412 may selectively fluidly couple to the coated particle melter 1050 and/or the crucible 428 via one or more dopant headers 414. The dopant distribution headers 414 may selectively fluidly couple to the coated particle melter 1050 and/or the crucible 428 via one or more dopant injectors 416. Prior to, during, or subsequent to the above explained charging phase, the dopant feed system 410 may selectively supply one or more contents of the dopant reservoir 412 to the coated particle melter 1050 and/or the crucible 428. The doping subsystem 410 advantageously removes any need to open the coated particle melter 1050 and/or the crucible 428.

Additionally or alternatively, an interior space of the crucible 428 may be completely or partially hermetically sealed from the interior space 1054 of the coated particle melter 1050. In such a case, each element explained above as fluidly coupling to the coated particle melter 1050 may fluidly couple to the interior space of the crucible 428.

The above description of illustrated embodiments, including what is described in the Abstract, is not intended to be exhaustive or to limit the embodiments to the precise forms disclosed. Although specific embodiments and examples are described above for illustrative purposes, various equivalent modifications can be made without departing from the spirit and scope of the disclosure, as will be recognized by those skilled in the relevant art. The teachings provided above of the various embodiments can be applied to other

systems, methods and/or processes for producing silicon and other materials (e.g., semiconductor materials, for instance SiGe), not only the exemplary systems, methods and devices generally described above.

For instance, the detailed description above has set forth various embodiments of the systems, processes, methods and/or devices via the use of block diagrams, schematics, flow charts and examples. Insofar as such block diagrams, schematics, flow charts and examples contain one or more functions and/or operations, it will be understood by those skilled in the art that each function and/or operation within such block diagrams, schematics, flowcharts or examples can be implemented, individually and/or collectively, by a wide range of system components, hardware, software, firmware, or virtually any combination thereof.

In certain embodiments, the systems used or devices produced may include fewer structures or components than in the particular embodiments described above. In other embodiments, the systems used or devices produced may include structures or components in addition to those described herein. In further embodiments, the systems used or devices produced may include structures or components that are arranged differently from those described herein. For example, in some embodiments, there may be additional heaters and/or mixers and/or separators in the system to provide effective control of temperature, pressure, or flow rate. Further, in implementation of procedures or methods described herein, there may be fewer operations, additional operations, or the operations may be performed in different order from those described herein. Removing, adding, or rearranging system or device components, or operational aspects of the processes or methods, would be well within the skill of one of ordinary skill in the relevant art in light of this disclosure.

The operation of methods and systems for making polysilicon described herein may be under the control of automated control systems. Such automated control systems may include one or more of appropriate sensors

(*e.g.*, flow sensors, pressure sensors, temperature sensors), actuators (*e.g.*, motors, valves, solenoids, dampers), chemical analyzers and processor-based systems which execute instructions stored in processor-readable storage media to automatically control the various components and/or flow, pressure and/or temperature of materials based at least in part on data or information from the sensors, analyzers and/or user input.

Regarding control and operation of the systems and processes, or design of the systems and devices for making polysilicon, in certain embodiments the present subject matter may be implemented via Application Specific Integrated Circuits (ASICs). However, those skilled in the art will recognize that the embodiments disclosed herein, in whole or in part, can be equivalently implemented in standard integrated circuits, as one or more computer programs running on one or more computers (*e.g.*, as one or more programs running on one or more computer systems), as one or more programs running on one or more controllers (*e.g.*, microcontrollers) as one or more programs running on one or more processors (*e.g.*, microprocessors), as firmware, or as virtually any combination thereof. Accordingly, designing the circuitry and/or writing the code for the software and or firmware would be well within the skill of one of ordinary skill in the art in light of this disclosure.

The various embodiments described above can be combined to provide further embodiments. Aspects of the embodiments can be modified, if necessary to employ concepts of various patents, applications and publications to provide yet further embodiments.

These and other changes can be made to the embodiments in light of the above-detailed description. In general, in the following claims, the terms used should not be construed to limit the claims to the specific embodiments disclosed in the specification and the claims, but should be construed to include all possible embodiments along with the full scope of equivalents to which such claims are entitled. Accordingly, the claims are not limited by the disclosure.

To the extent that they are not inconsistent with the specific teachings and definitions herein, all of the US patents, US patent application publications, US patent applications, referred to in this specification and/or
5 listed in the Application Data Sheet, including but not limited to U.S. Patent Application Serial No. 62/315,334, filed March 30, 2016, are incorporated herein by reference in their entirety.

CLAIMS

1. A dust suppression inducing crystal production method, the method comprising:

maintaining a high vacuum within a particulate feed vessel;

maintaining a high vacuum within a melting chamber;

fluidly connecting the particulate feed vessel and a crucible inside the melting chamber;

conveying silicon granules from the particulate feed vessel to the crucible inside the melting chamber;

melting the silicon granules in the crucible inside the melting chamber; and

charging the melted silicon under the maintained high vacuum to suppress dust particles during at least one of charging or re-charging of the crucible.

2. The method of claim 1, wherein conveying silicon granules from the particulate feed vessel to the crucible inside the melting chamber includes conveying silicon granules under a high vacuum from the particulate feed vessel to the crucible inside the melting chamber.

3. The method of claim 1, wherein conveying silicon granules from the particulate feed vessel to the crucible inside the melting chamber includes conveying silicon granules from the particulate feed vessel to the crucible inside the melting chamber via a conduit having an interior, and further comprising:

maintaining a high vacuum in the interior of the conduit at least while conveying silicon granules there along.

4. The method of any of claims 1 through 3, further comprising:

adding at least one dopant to at least one of the melting chamber or the crucible.

5. The method of claim 4 wherein adding at least one dopant includes adding at least one dopant in a particulate form.

6. The method of any of claims 1 through 5, further comprising:

purging gas in the melter during the charging or re-charging of the crucible while maintaining the high vacuum in the melter.

7. The method of any of claims 1 through 5, further comprising:

reducing an amount of gaseous purging in the melter during the charging or re-charging of the crucible to maintain the high vacuum.

8. The method of any of claims 1 through 5, further comprising:

foregoing any gaseous purging in the melter during the charging or re-charging of the crucible to maintain the high vacuum.

9. The method of any of claims 1 through 5, wherein the high vacuum is 300 Torr or less.

10. The method of any of claims 1 through 5, wherein the high vacuum is 200 Torr or less.

11. The method of any of claims 1 through 5, wherein the high vacuum is 100 Torr or less.

12. The method of any of claims 1 through 5, wherein the high vacuum is 20 Torr or less.

13. The method of any of claims 1 through 5, wherein the dust particles are 500 microns or less in diameter.

14. The method of any of claims 1 through 5, wherein the dust particles are 300 microns or less in diameter.

15. The method of any of claims 1 through 5, wherein the dust particles are 100 microns or less in diameter.

16. The method of any of claims 1 through 5, wherein the dust particles are 20 microns or less in diameter.

17. A dust suppression inducing crystal production system, the system comprising:

a particulate feed vessel;

a melting chamber containing a crucible;

a conduit that fluidly connects the particulate feed vessel and the crucible inside the melting chamber, the conduit through which silicon granules are conveyed from the particulate feed vessel to the crucible inside the melting chamber via a high vacuum environment during at least one of a charging or a re-charging of the crucible, the dust particles suppressed by the high vacuum as the granules are at least one of charged or re-charged into the molten silicon under the high vacuum,

18. The system of claim 17, further comprising:

a gas purging subsystem fluidly coupled to the melting chamber and operable to maintaining the high vacuum in the melting chamber.

19. The system of claim 17, further comprising:

a gas purging subsystem fluidly coupled to the melting chamber and operable to reduce an amount of gaseous purging in the melt chamber during the charging or re-charging of the crucible to maintain the high vacuum.

20. The system of claim 17, further comprising:

a gas purging subsystem fluidly coupled to the melting chamber and operable to forego any gaseous purging in the melter during the charging or re-charging of the crucible to maintain the high vacuum.

21. The system of claim 17, further comprising:

a doping subsystem fluidly coupled to the melting chamber and operable to add at least one dopant to at least one of the melting chamber or the crucible.

22. The system of claim 21 wherein adding at least one dopant includes adding at least one dopant in a particulate form.

23. The system of any of claims 17 through 22, wherein the high vacuum is 300 Torr or less.

24. The system of any of claims 17 through 22, wherein the high vacuum is 200 Torr or less.

25. The system of any of claims 17 through 22, wherein the high vacuum is 100 Torr or less.

26. The system of any of claims 17 through 22, wherein the high vacuum is 20 Torr or less.

27. The system of any of claims 17 through 26, wherein the dust particles are 500 microns or less in diameter.

28. The system of any of claims 17 through 26, wherein the dust particles are 300 microns or less in diameter.

29. The system of any of claims 17 through 26, wherein the dust particles are 100 microns or less in diameter.

30. The system of any of claims 17 through 26, wherein the dust particles are 20 microns or less in diameter.

31. A dust suppression inducing crystal production method, the method comprising:

maintaining a high vacuum within a particulate feed vessel and a melting chamber;

opening at least one valve to fluidly connect the particulate feed vessel and a crucible inside the melting chamber via a conduit;

conveying silicon granules from the particulate feed vessel to the crucible inside the melting chamber; and

melting the silicon granules in the crucible inside the melting chamber.

32. The method of claim 31, wherein conveying silicon granules from the particulate feed vessel to the crucible inside the melting chamber includes at least one of charging or re-charging the melted silicon in

the crucible under high vacuum during at least one of a charging or re-charging phase.

33. The method of claim 31, wherein conveying silicon granules from the particulate feed vessel to the crucible inside the melting chamber includes conveying silicon granules from the particulate feed vessel to the crucible inside the melting chamber via an interior of the conduit, and further comprising:

maintaining a high vacuum in the interior of the conduit at least while conveying silicon granules therealong.

34. The method of any of claims 31 through 33, wherein opening at least one valve to fluidly connect the particulate feed vessel and a crucible inside the melting chamber includes slowly opening the at least one valve to equalize pressure between the melting chamber and an interior of the conduit.

35. The method of any of claims 31 through 33, further comprising:

equalizing a pressure between an interior of the conduit and at least one of the particulate feed vessel or the inside of the melting chamber.

36. The method of any of claims 31 through 35, further comprising:

purging gas in the melter during the charging or re-charging of the crucible while maintaining the high vacuum in the melter.

37. The method of any of claims 31 through 35, further comprising:

reducing an amount of gaseous purging in the melter during the charging or re-charging of the crucible to maintain the high vacuum.

38. The method of any of claims 31 through 35, further comprising:

foregoing any gaseous purging in the melter during the charging or re-charging of the crucible to maintain the high vacuum.

39. The method of any of claims 31 through 38, further comprising:

adding at least one dopant to at least one of the charging chamber or the crucible.

40. The method of claim 39 wherein adding at least one dopant includes adding at least one dopant in a particulate form.

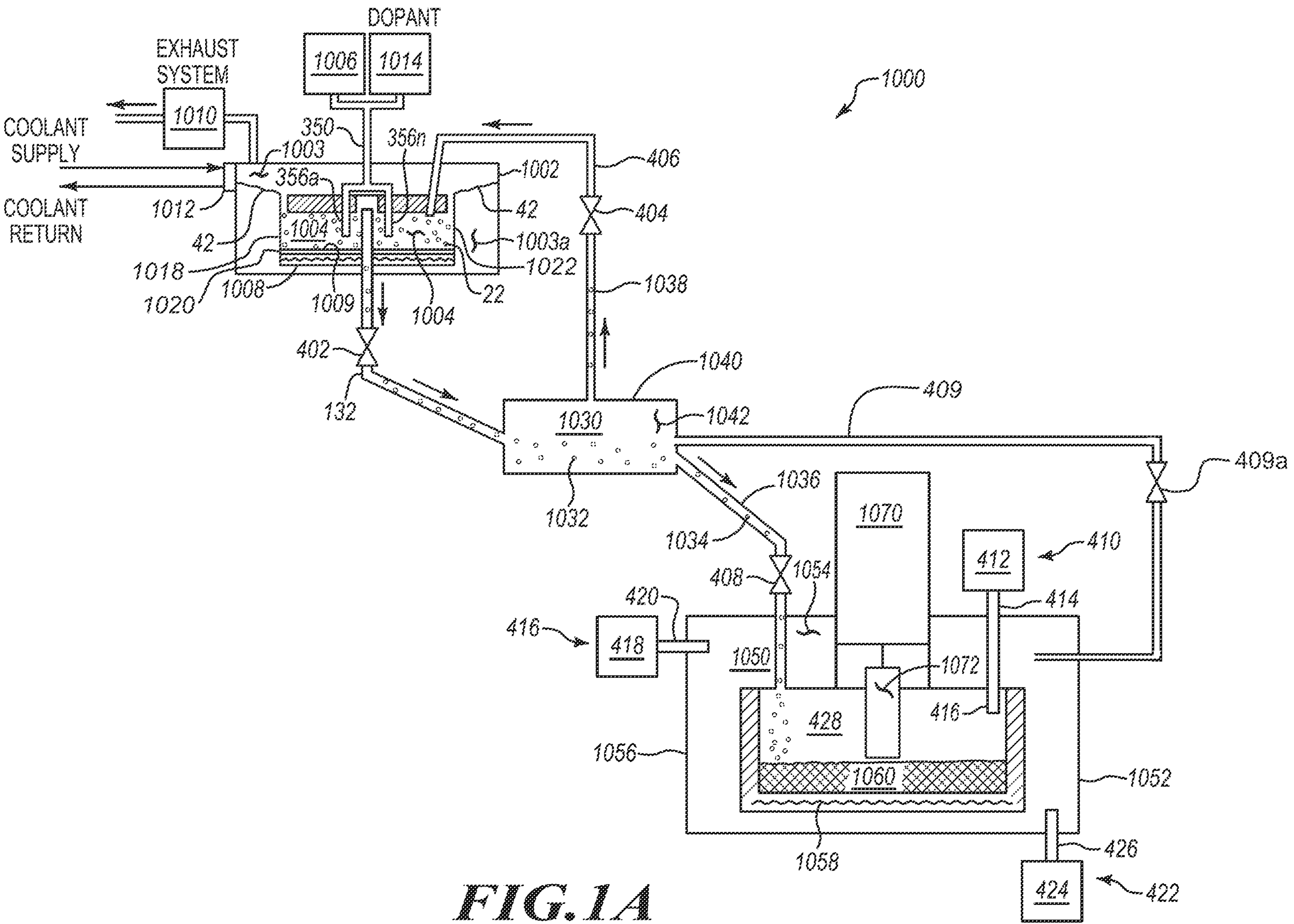

FIG. 1A

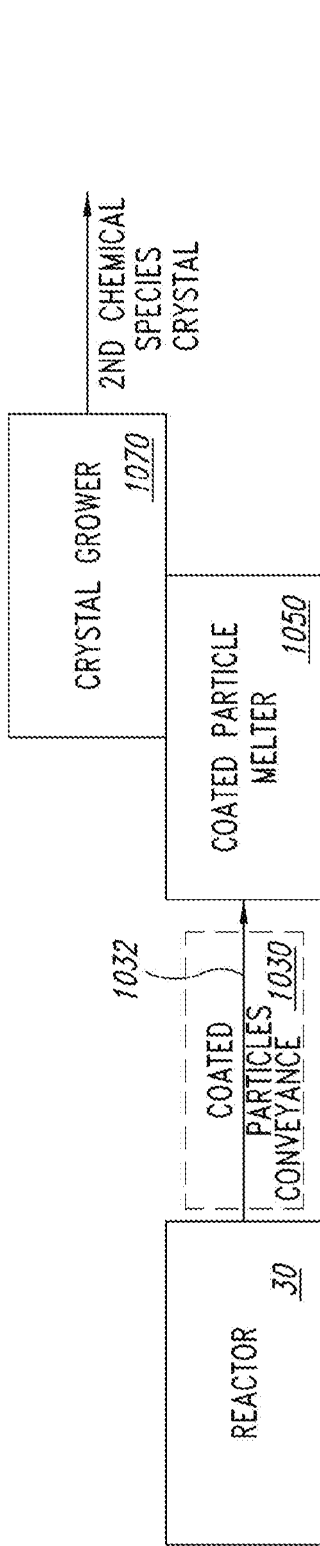

FIG. 1B

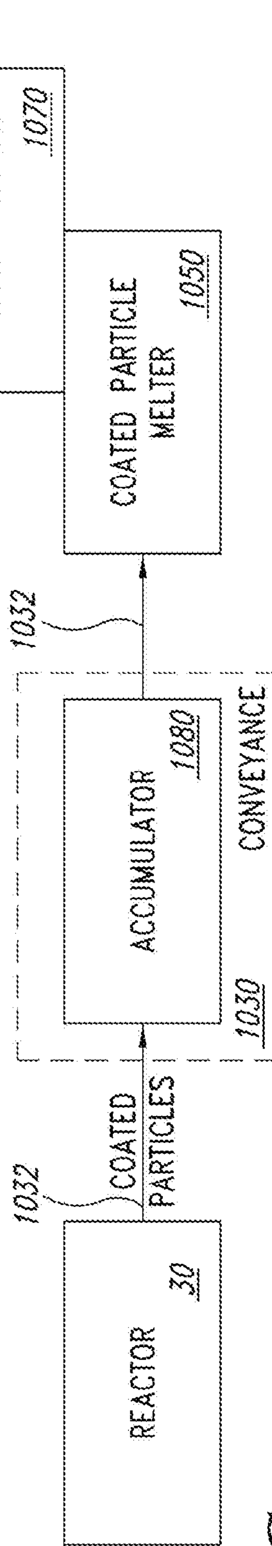

FIG. 1C

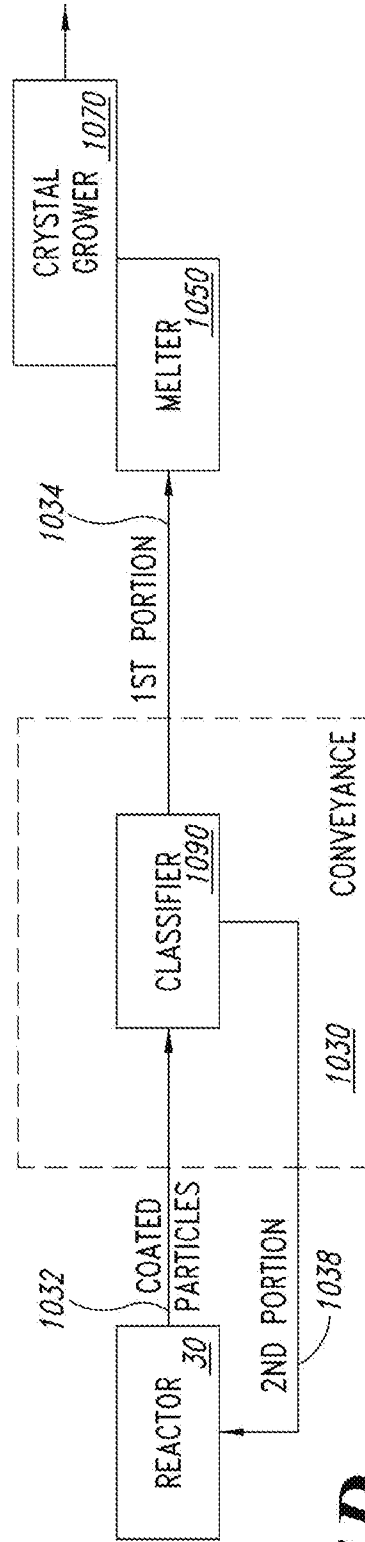

FIG. 1D

REACTOR 30

1032 COATED PARTICLES

ACCUMULATOR 1080

COATED PARTICLES

CLASSIFIER 1090

CONVEYANCE 1030

2ND PORTION 1038

1ST PORTION 1034

MELTER 1050

CRYSTAL GROWER 1070

FIG. 1E

REACTOR 30

1032 COATED PARTICLES

CLASSIFIER 1090

LARGE ø FRACTION 1092

SMALL ø FRACTION 1094

PARTICLE GRINDER 1096

CONVEYANCE 1030

2ND PORTION 1038

1ST PORTION 1034

MELTER 1050

CRYSTAL GROWER 1070

FIG. 1F

REACTOR 30

1032 COATED PARTICLES

ACCUMULATOR 1080

CLASSIFIER 1090

LARGE ø FRACTION 1092

SMALL ø FRACTION 1094

PARTICLE GRINDER 1096

CONVEYANCE 1030

2ND PORTION 1038

1ST PORTION 1034

MELTER 1050

CRYSTAL GROWER 1070

FIG. 1G

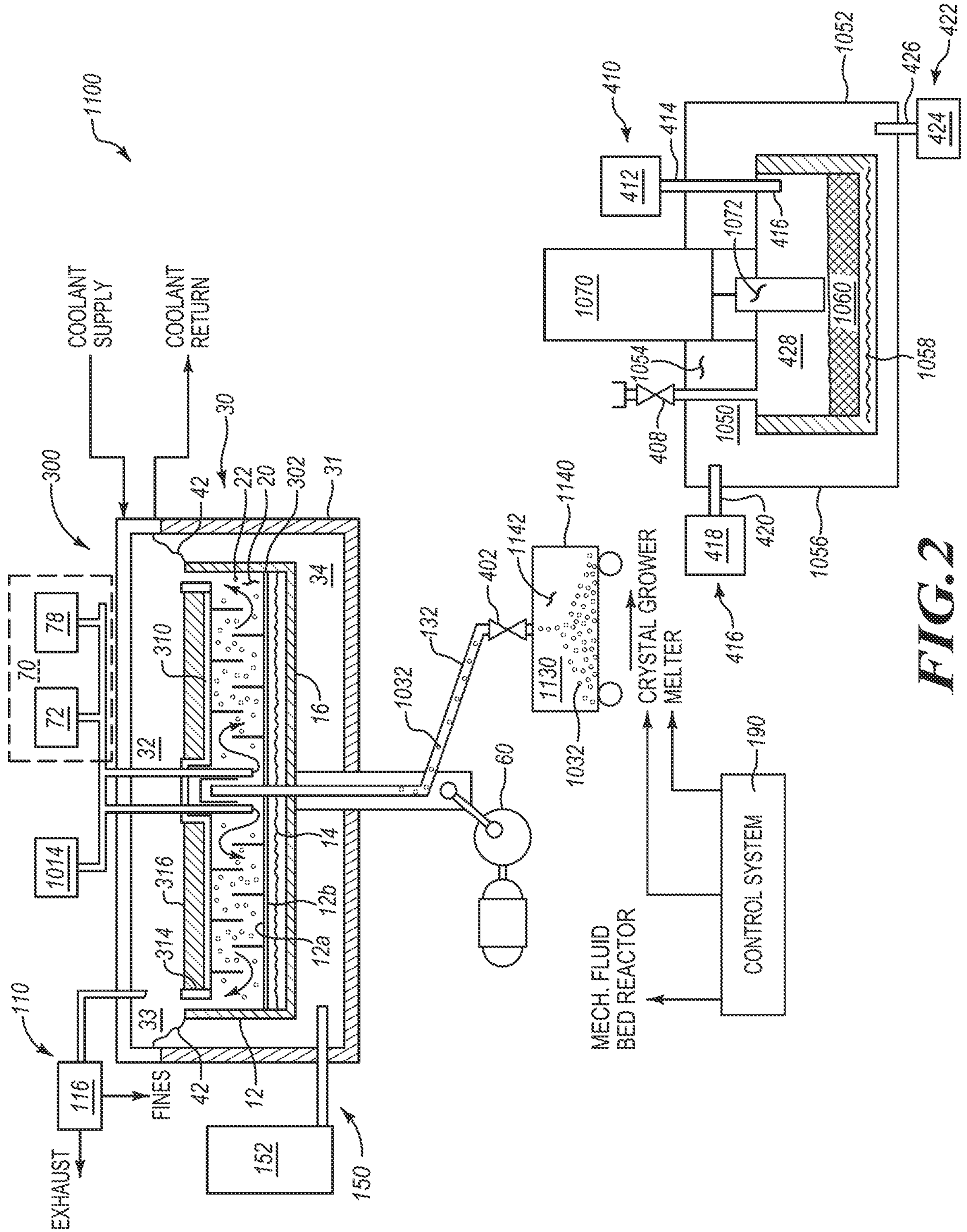

FIG. 2

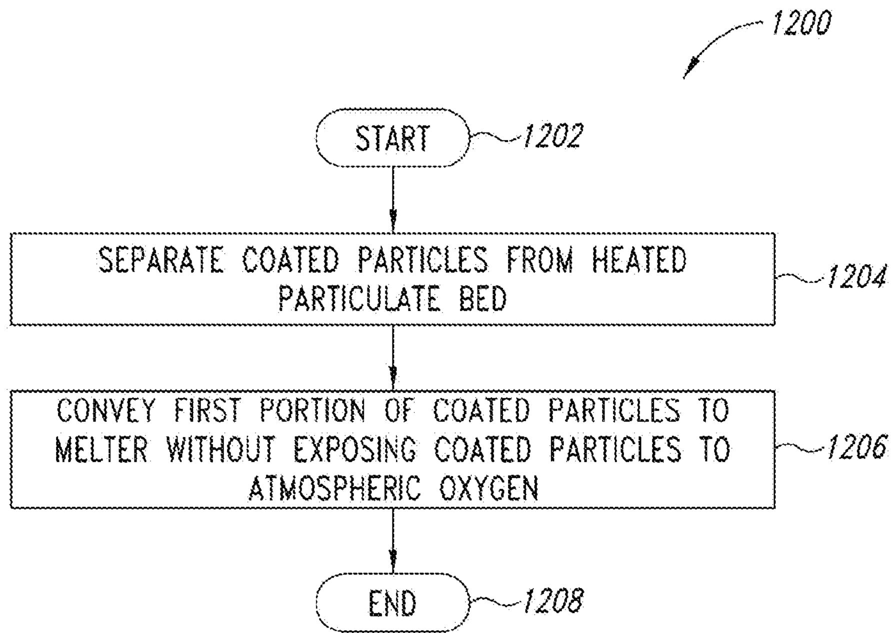

FIG. 3

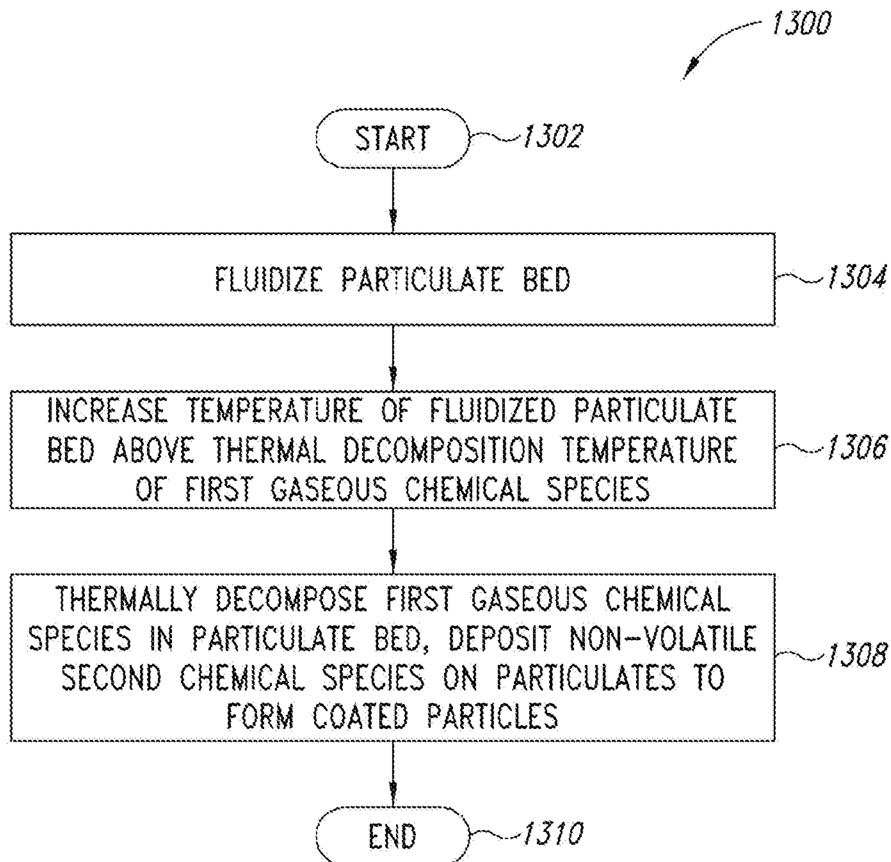

FIG. 4

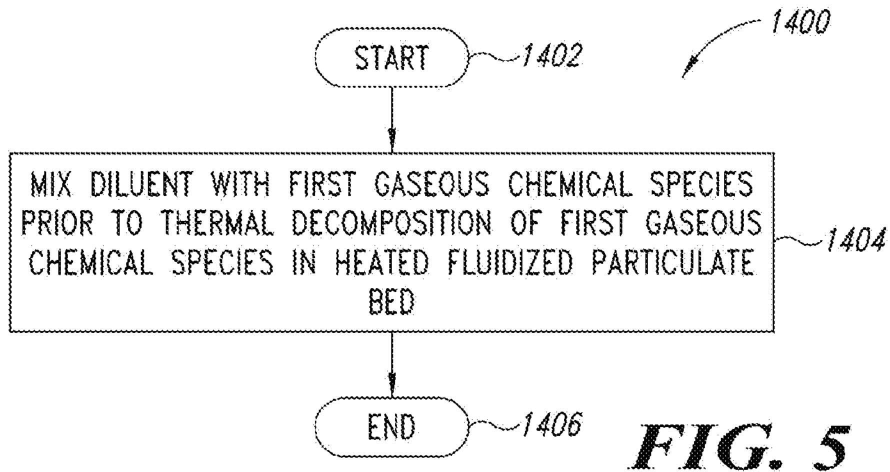

FIG. 5

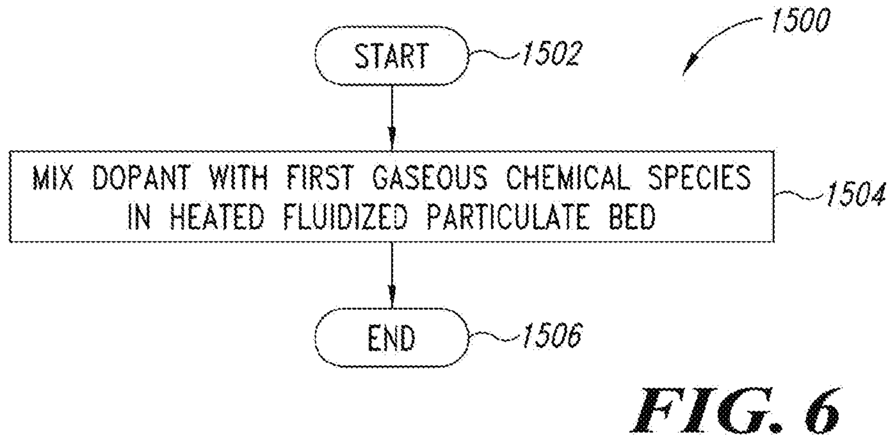

FIG. 6

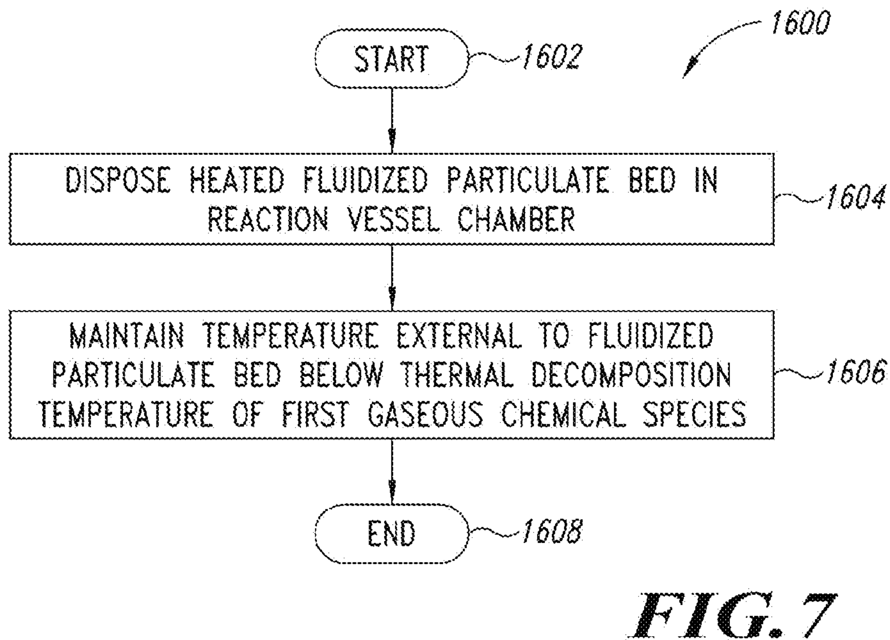

FIG. 7

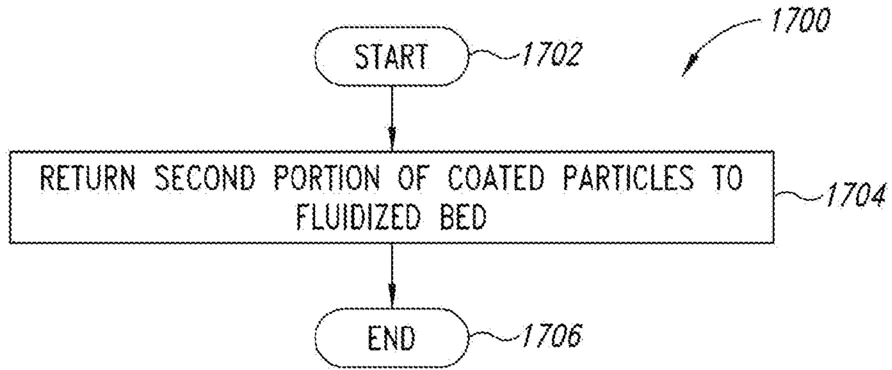

FIG. 8

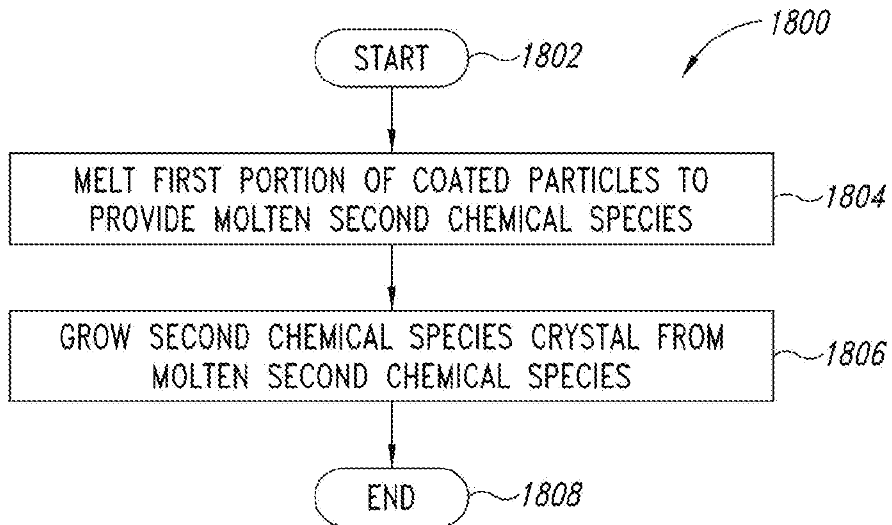

FIG. 9

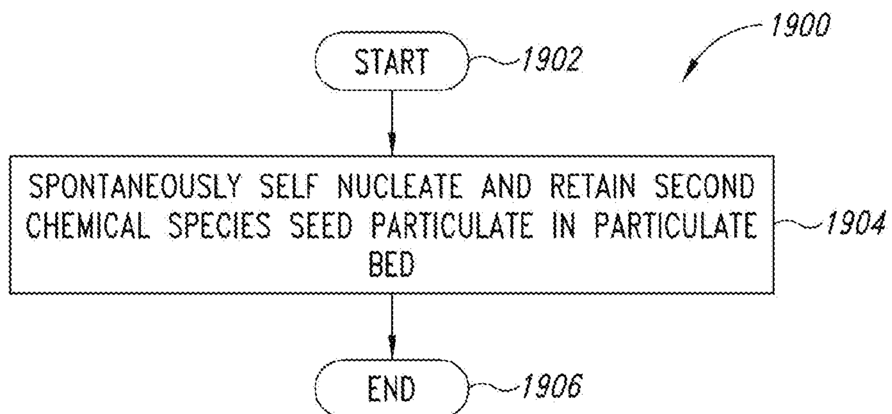

FIG. 10

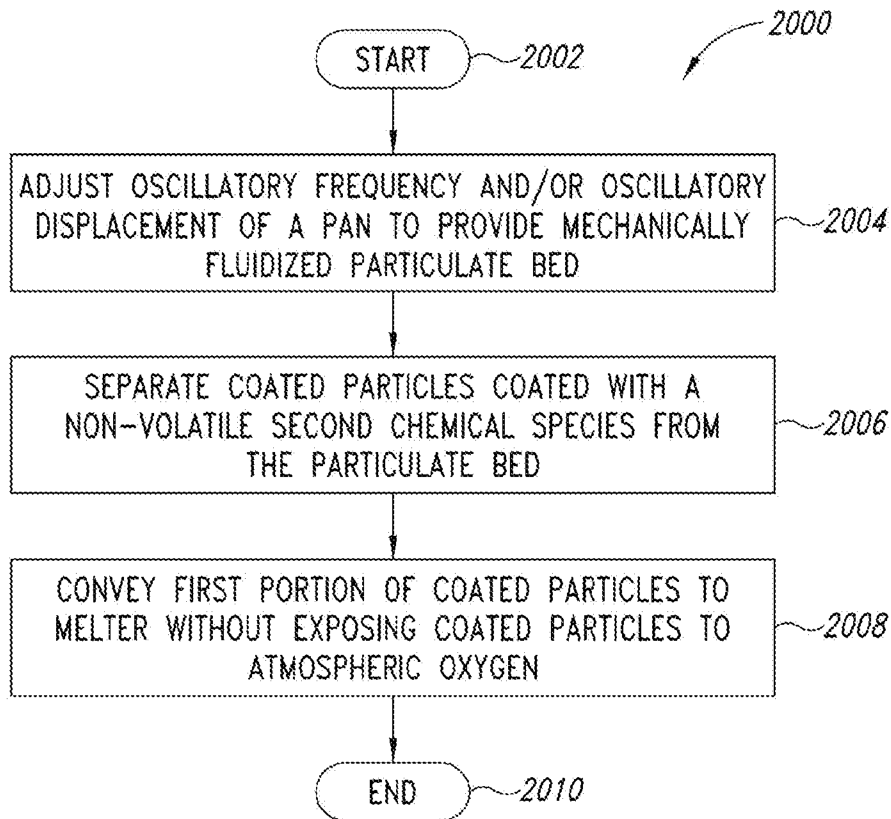

FIG. 11

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2017/024512

A. CLASSIFICATION OF SUBJECT MATTER

C01B 33/021(2006.01)i, B65G 53/04(2006.01)i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C01B 33/021; C30B 15/00; C03B 19/02; B65G 35/00; C30B 29/06; C30B 27/02; H01B 1/04; B65D 85/84; B65G 53/04

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Korean utility models and applications for utility models
Japanese utility models and applications for utility models

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
eKOMPASS(KIPO internal) & Keywords: silicon, granule, melting, crucible, vacuum pressure, solid dopant, dust supression

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2012-0159993 A1 (GEERTSEN, ROBERT J. et al.) 28 June 2012 See paragraphs [0003], [0014], [0038], [0039], [0048]-[0052], [0062], [0071]; and claims 1, 13.	1-3,17-20,23-26,31-35
Y		4,5,21,22
Y	US 2012-0056135 A1 (DELUCA, JOHN P. et al.) 08 March 2012 See paragraph [0014]; and claims 1, 15.	4,5,21,22
A	WO 2012-145250 A2 (GT ADVANCED CZ, LLC.) 26 October 2012 See paragraphs [0021]-[0030]; and claim 1.	1-5,17-26,31-35
A	US 2011-0024266 A1 (BAUMANN, BERNHARD et al.) 03 February 2011 See paragraphs [0009]-[0012]; and claims 1-4.	1-5,17-26,31-35
A	US 2005-0279275 A1 (HOLDER, JOHN DAVIS) 22 December 2005 See paragraphs [0051], [0070]-[0074]; and claim 1.	1-5,17-26,31-35

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents,such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
10 July 2017 (10.07.2017)	**11 July 2017 (11.07.2017)**

Name and mailing address of the ISA/KR
International Application Division
Korean Intellectual Property Office
189 Cheongsa-ro, Seo-gu, Daejeon, 35208, Republic of Korea
Facsimile No. +82-42-481-8578

Authorized officer
CHO, Ki Yun
Telephone No. +82-42-481-5655

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2017/024512

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☒ Claims Nos.: 40
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
Claim 40 refers to a multiple dependent claim which does not comply with PCT Rule 6.4(a).

3. ☒ Claims Nos.: 6-16, 27-30, 36-39
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required addtional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fees, this Authority did not invite payment of any additional fees.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.

☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.

☐ No protest accompanied the payment of additional search fees.

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2012-0159993 A1	28/06/2012	TW 201233466 A	16/08/2012
		WO 2012-074934 A1	07/06/2012
US 2012-0056135 A1	08/03/2012	CN 103249875 A	14/08/2013
		CN 103249875 B	12/10/2016
		EP 2611952 A2	10/07/2013
		EP 2611952 A4	22/01/2014
		JP 2013-539451 A	24/10/2013
		JP 5934218 B2	15/06/2016
		KR 10-2014-0021508 A	20/02/2014
		TW 201213626 A	01/04/2012
		TW I526584 B	21/03/2016
		US 9051659 B2	09/06/2015
		WO 2012-031136 A2	08/03/2012
		WO 2012-031136 A3	26/04/2012
WO 2012-145250 A2	26/10/2012	CN 103597127 A	19/02/2014
		EP 2699716 A2	26/02/2014
		JP 2014-512330 A	22/05/2014
		JP 6097276 B2	15/03/2017
		KR 10-2014-0097972 A	07/08/2014
		TW 201319333 A	16/05/2013
		US 2012-266808 A1	25/10/2012
		WO 2012-145250 A3	24/01/2013
US 2011-0024266 A1	03/02/2011	AT 513773 T	15/07/2011
		CA 2708997 A1	31/01/2011
		CA 2708997 C	27/11/2012
		CN 101987694 A	23/03/2011
		CN 101987694 B	19/08/2015
		DE 102009028166 A1	03/02/2011
		EP 2279970 A1	02/02/2011
		EP 2279970 B1	22/06/2011
		ES 2366017 T3	14/10/2011
		JP 2011-032100 A	17/02/2011
		JP 5394336 B2	22/01/2014
		KR 10-1242019 B1	11/03/2013
		KR 10-2011-0013229 A	09/02/2011
		US 8939277 B2	27/01/2015
US 2005-0279275 A1	22/12/2005	CN 101006205 A	25/07/2007
		CN 101006205 B	09/11/2011
		CN 101724903 A	09/06/2010
		CN 101724903 B	13/02/2013
		EP 1756337 A2	28/02/2007
		EP 1756337 B1	22/04/2009
		JP 2008-503427 A	07/02/2008
		JP 5080971 B2	21/11/2012
		KR 10-1300309 B1	28/08/2013

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/US2017/024512

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
		TW 200617220 A	01/06/2006
		TW I347377 B	21/08/2011
		US 2005-0279276 A1	22/12/2005
		US 2005-0279278 A1	22/12/2005
		US 7344594 B2	18/03/2008
		US 7465351 B2	16/12/2008
		US 7691199 B2	06/04/2010
		WO 2006-009802 A2	26/01/2006
		WO 2006-009802 A3	24/08/2006