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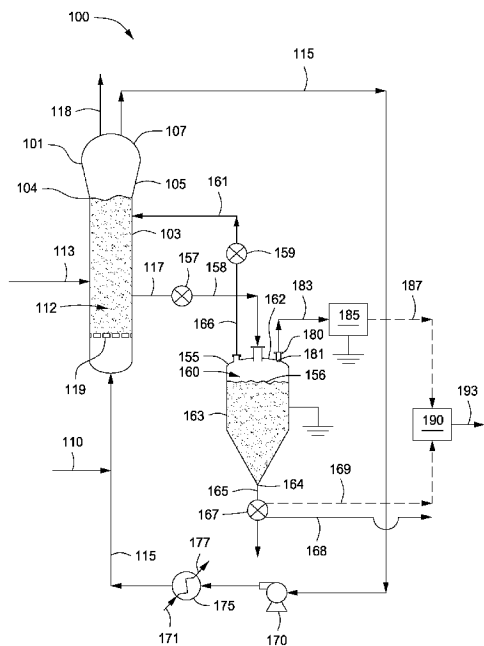


FIG. 1

(57) Abstract: Systems and methods for estimating a static charge on particulates are provided. The method includes measuring an electrical signal generated during an introduction of particulates to a discharge vessel or an electrical signal generated during removal of the particulates from the discharge vessel. The method also includes integrating the measured electrical signal. The method includes estimating a total charge (Q) on the particulates introduced to the discharge vessel at least in part by using the integrated electrical signal.



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SYSTEMS AND METHODS FOR MEASURING STATIC CHARGE ON PARTICULATES

BACKGROUND

[0001] In gas phase polymerization, a gaseous stream containing one or more monomers is passed through a fluidized bed under reactive conditions in the presence of a catalyst. A polymer product is withdrawn from the reactor, fresh monomer is introduced to the reactor to replace the removed polymer product, and any unreacted monomer is recycled back to the reactor. Process upsets in the reactor are often related to the buildup of catalyst and/or polymer particulates on the walls and/or other surfaces, *e.g.* distribution plate, within the reactor. The occurrence of catalyst and/or polymer buildup ("agglomerations") is often referred to as sheeting, chunking, drooling, or plugging. When this buildup becomes sufficiently large, fluidization can be disrupted, which can require the reactor to be shut down.

[0002] Numerous techniques are used to measure the amount of buildup and/or estimate the likelihood that buildup may occur within the reactor. One approach involves measuring static charge on the catalyst/polymer being produced within the reactor. The principal cause for static charge generation in the reactor is frictional contact of dissimilar materials by a physical process known as frictional electrification or the triboelectric effect. In gas phase polymerization reactors, the static is generated by frictional contact between the catalyst and polymer particulates and the reactor walls.

[0003] Conventional static charge measurement systems use static probes in communication with the reactor. These static probes, however, are only capable of measuring static charge present in localized areas within the reactor and cannot provide an indication of the total accumulated charge on the polymer product within the reactor. An attempt to overcome the limitations of the conventional static probes has been to modify a product discharge tank to include an insulated Faraday drum within the discharge tank. The Faraday drum can provide a quantitative measure as to the quantity of charge on the polymer product that is introduced to the discharge tank. Modified discharge tanks having a Faraday drum, however, have not been commercially implemented due to high costs and potential safety concerns.

[0004] There is a need, therefore, for improved systems and methods for estimating the static charge on a polymer product.

SUMMARY

[0005] Systems and methods for estimating a static charge on particulates are provided. The method can include measuring an electrical signal generated during an introduction of particulates to a discharge vessel or an electrical signal generated during removal of the

particulates from the discharge vessel. The method can also include integrating the measured electrical signal. The method can also include estimating a total charge (Q) on the particulates introduced to the discharge vessel at least in part by using the integrated electrical signal.

[0006] Another method for estimating a static charge on particulates can include introducing particulates from a polymerization reactor to an internal volume of a discharge vessel. The method can also include removing the particulates from the discharge vessel. The method can also include measuring a static probe electrical signal generated during the introduction of the particulates to the discharge vessel or a static probe electrical signal generated during the removal of the particulates from the discharge vessel. The measured electrical signal can be integrated. The integrated electrical signal can be multiplied by a proportionality constant (κ) to provide an estimated charge (Q) on the particulates introduced to the discharge vessel.

[0007] The system for estimating an electrical charge on particulates can include a discharge vessel having an internal volume for receiving particulates. A first conduit can be in fluid communication with the internal volume and adapted to introduce the particulates to the internal volume. A second conduit can be in fluid communication with the discharge vessel and adapted to remove the particulates from the internal volume. A static probe can be in communication with the internal volume and adapted to detect an electrical signal generated as the particulates are introduced to the internal volume, as the particulates are removed from the internal volume, or both. The system can also include an electrometer in communication with the static probe and adapted to measure the electrical signal detected by the static probe.

BRIEF DESCRIPTION OF THE DRAWINGS

[0008] Figure 1 depicts a schematic of an illustrative system for estimating a static charge on particulates introduced from a first vessel to a second vessel.

[0009] Figure 2 depicts a graphical depiction showing a current signal measured from a static probe and electrometer in communication with the internal volume of the discharge vessel as a polymer product was introduced to and removed from the discharge vessel.

[0010] Figure 3 depicts a close-up view of a portion of the graphical depiction shown in Figure 2.

DETAILED DESCRIPTION

[0011] Figure 1 depicts a schematic of an illustrative system 100 for estimating static charge on particulates introduced from a first or "particulate production" vessel 101 to a second or "discharge" vessel 155. At least a portion of particulates within the particulate production vessel 101 can be recovered via conduit or line 117. A first flow control device 157 can be used to control or otherwise adjust an amount of the particulates via conduit or line 158 introduced from

the particulate production vessel 101 to the discharge vessel 155. A second flow control device 167 can be used to control or otherwise adjust the rate at which the particulates via conduit or line 165 are removed from the discharge vessel 155.

[0012] One or more static probes (one is shown) 180 can be in communication with an internal volume 160 of the discharge vessel 155. Any number of static probes 180 can be in communication with the internal volume 160. For example one, two, three, four, five, or ten probes 180 can be in communication with the internal volume 160. The static probe 180 can be electrically insulated from the discharge vessel 155. The static probe 180 can include a probe tip 181 that extends into the internal volume 160 of the discharge vessel 155. In another example, the probe tip 181 can be flush with an inner surface of the discharge vessel 155. In still another example, the probe tip 181 can be recessed within the inner surface of the discharge vessel 155.

[0013] For simplicity and ease of description, the system 100 shown in Figure 1 will be discussed in the context of a gas phase polymerization system. As such, the particulate production vessel 101 can be referred to as a gas phase polymerization reactor or simply "reactor" 101, the discharge vessel 155 can be configured to receive a polymer product produced within the reactor 101, and the particulate product via line 158 can be referred to as the polymer product. However, it is noted that the systems and methods discussed and described herein can be applicable to a wide range of processes and are not limited to gas phase polymerization. Other illustrative processes can include, but are not limited to, slurry based polymerization systems, coal gasification, catalytic reforming, and cement production. Indeed, any process in which a static charge accumulates or could potentially accumulate on particulates can benefit from the systems and methods discussed and described herein.

[0014] It has been surprisingly and unexpectedly discovered that the static probe 180 in communication with the discharge vessel 155 can be monitored via one or more electrometers (one is shown) 185 to measure, estimate, or otherwise detect one or more electrical signals that can be used to determine or estimate a total accumulated charge on the particulates or "polymer product" introduced via line 158 thereto. For example, electrical signals generated as the polymer product via line 158 is introduced to the internal volume 160 of the discharge vessel 155, the "displacement signal," can be detected via the static probe 180 and the electrical signals can be communicated via line 183 to the electrometer 185. In another example, electrical signals generated as the polymer product via line 165 is removed from the internal volume 160 of the discharge vessel 155, the "rebound signal," can be measured, estimated, or otherwise detected via the static probe 180 and the electrical signals can be communicated via line 183 to the electrometer 185. The electrical signals measured using the static probe 180 and

electrometer 185 can include, but are not limited to, current and/or voltage. Accordingly, as used herein, the term "static probe" can include any probe or other device capable of being monitored via the electrometer 185 to measure, estimate, or otherwise detect one or more electrical signals, for example, current and/or voltage. The static probe 180 can detect the electrical signal(s) at any desired sampling rate. For example, the static probe 180 can detect the electrical signal(s) at a sampling frequency of about 90 Hz, about 100 Hz, about 125 Hz, about 150 Hz, or about 200 Hz or more than 200 Hz. Illustrative static probes 180 can include those discussed and described in U.S. Patent No. 6,008,662 and U.S. Patent Application Publication Nos. 2005/0148742, 2008/0319583, and 2009/0018279.

[0015] The static probe 180 can also be coated with one or more materials. Coating the static probe 180 can improve the quality of data via line 183 detected using the static probe 180. The static probe 180 can be coated with one or more polymers or polymer containing materials. Illustrative coating materials can include, but are not limited to, polytetrafluoroethylene (PTFE), perfluoroalkoxy (PFA), fluorinated ethylene propylene (FEP), or any combination thereof. The coating material can include XYLAN®, commercially available from Whitford Corporation. The coating can have a thickness ranging from a low of about 10 μm , about 50 μm , about 100 μm , or about 150 μm to a high of about 200 μm , about 300 μm , about 500 μm , or about 1,000 μm .

[0016] The electrometer 185 can be or include any system, device, or combination of systems and/or devices capable of measuring, estimating, or otherwise detecting the one or more electrical signals detected via the static probe 180 and communicated via line 183 thereto. An electrometer 185 that detects a flow of current from the probe tip 181 to ground can include, but is not limited to, an ammeter, a picoammeter (a high sensitivity ammeter), or a multi-meter. In another example, the electrometer 185 could also detect the current flow indirectly by measuring or estimating a voltage generated as the current flows through a resistor.

[0017] One or more processors (one is shown) 190 can be in communication via line 187 with the electrometer 185. The processor 190 can be programmed or otherwise configured to receive an output, *i.e.* the detected electrical signal, via line 187 from the electrometer 185 and process or manipulate the output data to estimate a charge on the polymer product via line 158 introduced to the discharge vessel 155 and/or a specific charge (*e.g.* $\mu\text{C/kg}$) on the polymer product introduced to the discharge vessel 155. The estimated charge on the polymer product can be output via line 193 to a display such as a monitor, an alarm, an automated control system, or the like.

[0018] The electrometer 185 can be used to measure an electrical signal, *e.g.*, current or "displacement current," detected via the static probe 180 and communicated via line 183 thereto that is generated as the charged polymer product via line 158 is introduced to the discharge

vessel 155. The electrical charge that enters the discharge vessel 155 with the polymer product causes a "displaced" charge to flow from a tip 181 of the probe 180, through the electrometer 185, and to ground. The displacement current results from the physical requirement that the grounded walls of the discharge vessel 155 and the probe tip 181 need to remain at zero potential, *i.e.* zero voltage relative to ground. Current can flow from the probe tip 181 to ground until the discharge vessel 155 is filled to a desired level with the polymer product and introduction of the polymer product via line 158 is stopped. Once introduction of the polymer product via line 158 is stopped, the current measured via the probe tip 181 goes to zero since no additional charged polymer product via line 158 is being introduced to the discharge vessel 155. The sign of the displacement current that exists during introduction of the polymer product to the discharge vessel 155 is the same as the net charge on the polymer product. For example, if the polymer product is positively charged, the displacement current is also positive. In other words, the positive charge flows from the probe tip 181 to ground or negative charges flow from ground, through the electrometer 185, to the probe tip 181.

[0019] The displacement current signal acquired during introduction of the polymer product via line 158 can be introduced as the output data via line 187 to the process 190. The processor 190 can integrate the output data over the period of time that the polymer product via line 158 was introduced to the discharge vessel 155 and provide the output via line 193. In another example, the processor 190 can receive the output data via line 187 in real time, *i.e.* as the polymer product via line 158 is introduced to the discharge vessel 155, and can integrate the data as it is received and provide the output via line 193 in real time.

[0020] The integrated displacement current provides a value that can be multiplied by a proportionality constant (κ) to yield the total charge on the polymer product introduced via line 158 to the discharge vessel 155. The proportionality constant (κ) is a constant unique for a particular discharge vessel 155 that is at least partially based on the particular dimensions, *e.g.* surface area and surface geometry of the inner walls of the discharge vessel 155, location of the probe 180, level 156 of polymer particulates introduced to the discharge vessel 155, and dimensions of the probe 180. The proportionality constant (κ) will be discussed and described in more detail below. The formula for determining the total charge on the polymer product introduced via line 158 to the discharge vessel 155 can be expressed by Equation 1:

$$Q = \kappa \int_{fill} i(t) dt \quad (\text{Equation 1})$$

where Q is the total charge (in units of Coulombs) on the polymer product introduced via line 158 to the discharge vessel 155; $\int_{fill} i(t)dt$ is the integrated current measured via the probe tip 181 over the time period that the polymer product via line 158 is introduced to the discharge vessel 155; and κ is the proportionality constant.

[0021] The polymer product via line 158 can be introduced at any desired rate. For example, typical gas phase polymerization systems introduce about 1,800 kg of polymer product per product discharge cycle to the discharge vessel 155 in a time of about 20 second to about a minute, which corresponds to about 30 kg/sec to about 90 kg/sec. However, any polymer product introduction rate ranging from about 1 kg/sec to about 200 kg/sec, or from 10 kg/sec to 180 kg/sec, or from 20 kg/sec to 100 kg/sec, for example, can be used.

[0022] After introducing the polymer product via line 158 to the discharge vessel 155, the pressure within the discharge vessel 155 can be at or close to a pressure within the polymerization reactor 101. In a typical gas phase polymerization system 100, the pressure within the discharge vessel 155, after introducing the polymer product via line 158 thereto, can range from 2,000 kPa to about 2,500 kPa. In one or more embodiments, the pressure within the discharge vessel 155 can be reduced by venting at least a portion of any fluid via line 166 from the discharge vessel 155. Venting the fluid via line 166 can be controlled by one or more flow control devices 159. The vented fluid can be recycled via line 161 to the polymerization reactor 101. During venting the displacement current from the probe tip 181 remains at zero because there is no electrical charge entering or leaving the discharge vessel 155. Venting at least a portion of the fluid via line 166 from the discharge vessel 155 can provide a pressure therein of from 350 kPa to about 1,400 kPa, for example.

[0023] The polymer product via line 165 can be removed from the discharge vessel 155 after introduction to the discharge vessel 155, with or without carrying out the optional venting operation. Removal of the polymer product via line 165 can be controlled by the one or more flow control devices 167. Similar to the polymer introduction, the polymer product via line 165 can be removed at any desirable rate. For example, typical gas phase polymerization systems remove the polymer product in about 50 second to about 2 minutes, which corresponds to about 15 kg/sec to about 36 kg/sec. However, any polymer product removal rate ranging from about 1 kg/sec to about 200 kg/sec, or 5 kg/sec to 150 kg/sec, or 10 kg/sec to 75 kg/sec, for example, can be used.

[0024] The electrometer 185 can measure an electrical signal, *e.g.*, current or "rebound current" detected via the static probe 180 and communicated via line 183 thereto that is generated when the charged polymer product via line 165 is removed from the discharge vessel 155. The

electrical charge that leaves the discharge vessel 155 with the polymer product causes a "rebound" charge to flow from ground, through the current meter 185, and back to the probe tip 181. Similar to the displacement current, the rebound current results from the physical requirement that the grounded walls of the discharge vessel 155 and the probe tip 181 need to remain at zero potential, *i.e.* zero voltage relative to ground. Current can continue to flow from ground to the probe tip 181 until removal of the polymer product via line 165 from the discharge vessel 155 is stopped. Once removal of the polymer product via line 165 is stopped, the current measured via the probe tip 181 goes to zero since no additional charged polymer product via line 165 is being removed from the discharge vessel 155. The sign of the rebound current is opposite to the sign of the displacement current and opposite the sign of the charge on the polymer product. For example, if the polymer product is positively charged, the rebound current is negative.

[0025] The rebound current signal detected by the electrometer 185 during introduction of the polymer product via line 158 can be introduced as the output, *i.e.* the detected electrical signal, via line 187 to the process 190. The processor 190 can integrate the detected electrical signal over the period of time that the polymer product via line 165 was removed from the discharge vessel 155 and provide the output via line 193. In another example, the processor 190 can receive the output data via line 187 in real time, *i.e.* as the polymer produce via line 165 is removed from the discharge vessel 155, and can integrate the data as it is received and provide the output via line 193 in real time.

[0026] The rebound current signal acquired during the time the polymer product via line 165 exits the discharge vessel 155 and output via line 187 to the process or 190 can be integrated. The integrated rebound current can provide a value that can be multiplied by the negative of the proportionality constant (κ) to yield the total charge on the polymer product introduced via line 158 to the discharge vessel 155. The formula for determining the total charge on the polymer product introduced via line 158 as the polymer product is removed from the discharge vessel 155 can be expressed by Equation 2:

$$Q = -\kappa \int_{discharge} i(t)dt \quad (\text{Equation 2})$$

[0027] where Q is the total charge (in units of Coulombs) of the polymer product introduced via line 158 to the discharge vessel 155; $\int_{discharge} i(t)dt$ is the integrated current measured via the probe tip 181 over the time period the polymer product is removed via line 165 from the discharge vessel 155; and κ is the proportionality constant.

[0028] Typical current levels measured via the static probe 180 and the electrometer 185 can range from about $\pm 0.1 \text{ nA/cm}^2$ to about $\pm 10 \text{ nA/cm}^2$, or about $\pm 0.1 \text{ nA/cm}^2$ to about $\pm 8 \text{ nA/cm}^2$, or about $\pm 0.1 \text{ nA/cm}^2$ to about $\pm 4 \text{ nA/cm}^2$.

[0029] The processor 190 can be alerted or notified as to when the flow control device 167 is opened and closed in order to remove the polymer product from the discharge vessel 155. A time signal associated with opening and closing the flow control device 167 can be communicated to the processor 190 via line 169. Although not shown, the processor 190 could also be alerted or notified as to when the flow control device 157 and/or 159 are opened and closed. In another example, the processor 190 can determine when the flow control device(s) 157, 159, and/or 167 open and close based on the sampled data received via line 187 from the electrometer 185. As such, the need for a time signal associated with the opening and closing of the flow control device(s) 157, 159, and/or 167 can be avoided. In still another example, the processor 190 can determine when the flow control device(s) 157, 159, and/or 167 open and close based on a combination of the time signal via line 169 and the sampled data via line 187 received from the electrometer 185.

[0030] Either Equation 1 (integration of the displacement current multiplied by the proportionality constant (κ)) or Equation 2 (integration of the rebound current multiplied by the negative proportionality constant ($-\kappa$)) can be used to estimate the total charge on the polymer product introduced via line 158 to the discharge vessel 155. Using Equation 2 can be preferred over Equation 1 due to an increased probability of noise in the detected electrical signal being acquired during introduction of the polymer product via line 158 versus removal of the polymer product via line 165.

[0031] Not wishing to be bound by theory, it is believed that during introduction of the polymer product via line 158 to the discharge vessel 155 a significant amount of "noise" is generated due to physical contact between the polymer product and the probe tip 181. One possible reasons for increased noise during introduction of the polymer product relative to removal of the polymer product is that during normal operation there is usually a pressure differential between the polymer product and the internal volume 160 of the discharge vessel. For example, the polymer product can be at a pressure of about 2,000 kPa to about 2,500 kPa, but the internal volume 160 is only at a pressure of about 350 kPa to about 1,400 kPa. As such, it is believed that the pressure differential causes the polymer product to enter the internal volume 160 of the discharge vessel 155 and swirl, which causes some of the polymer product particulates to contact the probe tip 181. Contact between the probe tip 181 and the polymer product particulates cause a triboelectric charge transfer and it is believed that this triboelectric charge transfer is the source of noise detected via the static probe 180. Accordingly, it can be desirable

to isolate or protect or otherwise locate the probe tip 181 within the internal volume 160 of the discharge vessel 155, such that contact between polymer product and the probe tip 181 is minimized.

[0032] In one or more embodiments, the specific charge (q) on the polymer product rather than the total charge (Q) on the polymer product introduced to the discharge vessel 155 can be estimated or otherwise determined. The specific charge (q) when estimated via Equation 2, *i.e.* using the rebound current, can be expressed by Equation 3:

$$q = -\frac{Q}{M} = -\frac{\kappa}{M} \int_{discharge} i(t) dt \quad (\text{Equation 3})$$

[0033] where q is the specific charge and is defined as the charge per unit mass of polymer product in units of coulombs/kg (C/kg) or more commonly as micro-coulombs/kg ($\mu\text{C/kg}$); M is the mass of the polymer product introduced to the discharge vessel 155, and κ is the proportionality constant.

[0034] Integrating the charge measured via the probe 180 and electrometer 185 provides data as to the charge on a small portion of the total charge on the polymer product introduced via line 158 to the discharge vessel 155. As such, the electrical signal detected via the static probe 180 only represents a portion of the charge within the discharge vessel 155, *i.e.* the area represented by the probe tip 181 as compared to the area of the probe tip plus the inner surface area of the discharge vessel 155. Thus, it is the proportionality constant (κ) that facilitates estimating the charge on the entire polymer product introduced via line 158 to the discharge vessel 155.

[0035] The proportionality constant (κ) used in Equations 1-3, can be estimated, calculated, simulated, or otherwise determined using any suitable method or combination of methods. One method for determining the proportionality constant (κ) can be referred to as the "Area Ratio" method. Determining the proportionality constant (κ) using the Area Ratio method can be expressed by Equation 4.

$$\kappa = \frac{\text{Internal Surface Area of the Discharge Vessel}}{\text{Surface Area of the Probe Tip}} \quad (\text{Equation 4})$$

[0036] For example, a probe tip 181 having a surface area of 7.8 cm^2 and a discharge vessel 155 having an inner surface area of $1.77 \times 10^5 \text{ cm}^2$ would have a surface area ratio of about 22,700, as estimated via Equation 4. Equation 4 provides an estimate of the proportionality constant (κ) that falls within an order of magnitude of the correct or actual value. The displacement current (and the rebound current) detected via the static probe 180 and electrometer 185 can be less (for a given amount of charge) than for a typical point on the walls of the discharge vessel 155. As such, the Area Ratio method according to Equation 4 typically yields a proportionality constant

(κ) having a smaller value than the correct value. Without wishing to be bound by theory, it is believed that the reason for the discrepancy between proportionality constant (κ) determined using the Area Ratio method as compared to the actual value can be attributed to the fact that the probe tip 181 (as depicted in Figure 1) is more distant from the polymer product in the internal volume 160 than is a typical point on the walls of the discharge vessel 155. However, the Area Ratio method can provide a proportionality constant (κ) value that can be used in Equations 1-3 to provide a useful estimate of the total charge on the polymer product introduced via line 158 to the discharge vessel 155. In other words, the Area Ratio method can provide sufficient information that can be used by operation personnel and/or automated control systems in operating the polymerization reactor 101.

[0037] Another method that can be used for determining the proportionality constant (κ) can be referred to as the "Experimental" method. The Experimental method can include operating the static probe 180 under actual conditions, *i.e.* carrying out polymerization using the polymerization system 100 and introducing polymer product via line 158 to the discharge vessel 155, and measuring the specific charge on a particular sample of polymer product withdrawn from the discharge vessel 155. Such a measurement can be performed using a conventional Faraday cup apparatus, for example. An illustrative Faraday cup apparatus for measuring the static charge on a sampled portion of the polymer product from the discharge vessel 155 can be as discussed and described in U.S. Patent No. 6,686,743. The polymer product can be withdrawn via line 165, a sampling port (not shown), or other access point. The measurement of the particular sample of polymer product can then be multiplied by the total mass of the polymer product introduced to the discharge vessel 155. This calculation can provide an estimate of the total charge (Q) on the polymer product within the discharge vessel 155. The total charge (Q) can then be compared to the computed integral of either the displacement current or the rebound current, for example. The proportionality constant (κ) can then be estimated from the ratio of the measured charge (Q) to the estimated integrated displacement current or rebound current. For example, the Experimental method in which the integrated rebound current is used can be expressed by Equation 5:

$$\kappa = - \frac{\text{Estimated Total Charge } (Q)}{\int_{\text{discharge}} i(t) dt} \quad (\text{Equation 5})$$

[0038] Still another method for determining the proportionality constant (κ) can be referred to as the "Computational" or "Simulated" method. Calculations can be performed to determine or estimate the electric fields and resulting displacement and rebound currents as a function of a polymer product level 156 within the internal volume 160 of the discharge vessel 155. The

polymer product level 156 can be varied in the calculations to simulate the effect of an increasing and/or decreasing polymer product level 156 within the discharge vessel 155. These calculations can produce an estimate of the displacement current and/or rebound current that would be expected from a probe tip 181 having a given surface area and a given specific charge on the polymer product. The integrated values of the displacement current and/or rebound current can then be compared to the assumed total charge in the discharge vessel 155 to provide an estimate of the proportionality constant (κ).

[0039] If linear equations are used in the Computational method, a benefit can be that a computed proportionality constant (κ) for one particular level or amount of specific charge (q), e.g. 1 $\mu\text{C/kg}$, can provide results that apply for all levels or amounts of specific charge (q). Also, a series of computed proportionality constant (κ) values can be generated for different or varying polymer product levels 156 within the discharge vessel 155. The time derivative of this function for an assumed rate of polymer product introduction can provide the computed current that should be observed by the probe current versus time. The detected or measured current can also be integrated to give the charge detected via the probe tip 181 versus time, which can then be compared to the computed probe charge versus a polymer product level 156 within the discharge vessel 155.

[0040] Any method can be used to perform the computations required in the Computational method. For example, the computations can be carried out via a computer using appropriate software. For example, the geometry of the discharge vessel 155 can be acquired using Acrobat Reader from a file of engineering drawings and imported into GraphClick. GraphClick can take the locations of a sequence of points defining the outline of the discharge vessel 155. These values can be used to construct a drawing of the outline of the discharge vessel 155, including the location of the static probe 180, in COMSOL33. COMSOL33 is a software program that can be used to generate finite element meshes and numerical solutions of partial differential equations ("PDEs"). Other software programs that can be used to perform one or more of the computations used in the Computational method can include, but are not limited to, Abaqus, ADINA, ANSYS Multiphysics, CFD-ACE+, CFD-FASTAN, COMSOL Multiphysics, FlexPDE, LS-DYNA, NEi Nastran, CheFEM, Elmer, and OOFELIE.

[0041] In the COMSOL33 calculations, axial symmetry can be used. As such, the area over which the induced charge density is integrated can be simulated as an annular ring and the desired result can be obtained by scaling that area to that of the probe tip 181. The numerical calculations of a finite angle of repose for the polymer product level introduced the discharge vessel can be accounted for or disregarded. Additionally, an inverted conical profile for the

surface of the polymer product as the polymer product is removed from the discharge vessel 155 can be accounted for or disregarded.

[0042] Varying the polymer product level 156 within the internal volume 160 of the discharge vessel 155 between subsequent introduction and removal operations can cause the proportionality constant (κ) to change. As such, it can be desirable to maintain the amount of polymer product via line 158 introduced to the discharge vessel 155 at a constant or substantially constant amount, *e.g.* within about $\pm 5\%$, between subsequent cycles. Introducing the same or substantially the same amount of polymer product via line 158 to the discharge vessel 155 between subsequent runs can reduce or eliminate the need to re-estimate the proportionality constant (κ) for different polymer product discharge cycles. In another example, multiple proportionality constant (κ) values can be estimated for multiple polymer product levels 156 within the discharge vessel 155. Having proportionality constants (κ) for multiple polymer product levels 156 can reduce the need to introduce a constant or substantially constant amount of polymer product via line 158 to the discharge vessel 155 in subsequent polymer product discharge operations or cycles.

[0043] The static probe 180 can be disposed through a wall of the discharge vessel 155 at any location about the discharge vessel 155. As shown, the static probe 180 is disposed on a first end 162 of the discharge vessel 155. In another example, the static probe 180 can be disposed on a sidewall 163 of the discharge vessel 155. In still another example, the static probe 180 can be disposed on a sidewall 163 toward or at a second end 164 of the discharge vessel 155.

[0044] The polymer product via line 158 can be introduced to the internal volume 160 of the discharge vessel 155 from any location about the discharge vessel 155. As shown, the polymer product via line 158 is introduced to the internal volume 160 from the first end 162 of the discharge vessel 155. In another example, the polymer product via line 158 can be introduced to the internal volume 160 through the side wall 163 of the discharge vessel 155.

[0045] The probe tip 181, as noted above, can extend into the internal volume 160, be flush with the inner wall of the discharge vessel 155, or be recessed within the wall of the discharge vessel 155. If the probe tip 181 extends into the internal volume 160, the probe tip can extend any desired distance into the internal volume 160. For example, the probe tip 181 can extend a distance ranging from a low of about 1 mm, about 1 cm, or about 2 cm to a high of about 5 cm, about 15 cm, about 30 cm, or about 50 cm.

[0046] Preferably the static probe 180 and line 158 through which the polymer product is introduced to the internal volume 160 can be disposed about the discharge vessel 155, with respect to one another, such that direct contact between the polymer product and the probe tip 181 is minimized or reduced. For example, as shown in Figure 1, the polymer product is

introduced to the internal volume 160 from the first end 162 of the discharge vessel 155 and the static probe 180 is disposed through the first end 162 of the discharge vessel 155. Such an arrangement can minimize or reduce contact between the polymer particulates and the probe tip 181 because the polymer particulates are directed toward the second end 164 of the discharge vessel 155. As such, triboelectric charge generated during introduction of the polymer product via line 158 to the discharge tank 155 due to contact between the polymer product and the probe tip 181 can be reduced, thus, reducing the amount of noise generated in electrical measurements acquired via the static probe 180. The static probe 180 can also be shielded from the outside environment. Shielding the static probe 180 can reduce or eliminate noise from outside sources.

[0047] The polymer product(s) produced in the reactor 101 can be or include any type of polymer or polymeric material. For example, the polymer product can include homopolymers of olefins (*e.g.*, homopolymers of ethylene), and/or copolymers, terpolymers, and the like of olefins, particularly ethylene, and at least one other olefin. Illustrative polymers can include, but are not limited to, polyolefins, polyamides, polyesters, polycarbonates, polysulfones, polyacetals, polylactones, acrylonitrile-butadiene-styrene polymers, polyphenylene oxide, polyphenylene sulfide, styrene-acrylonitrile polymers, styrene maleic anhydride, polyimides, aromatic polyketones, or mixtures of two or more of the above. Suitable polyolefins can include, but are not limited to, polymers comprising one or more linear, branched or cyclic C₂ to C₄₀ olefins, preferably polymers comprising propylene copolymerized with one or more C₃ to C₄₀ olefins, preferably a C₃ to C₂₀ alpha olefin, more preferably C₃ to C₁₀ alpha-olefins. More preferred polyolefins include, but are not limited to, polymers comprising ethylene including but not limited to ethylene copolymerized with a C₃ to C₄₀ olefin, preferably a C₃ to C₂₀ alpha olefin, more preferably propylene, butene, and/or hexene.

[0048] Preferred polymers include homopolymers or copolymers of C₂ to C₄₀ olefins, preferably C₂ to C₂₀ olefins, preferably a copolymer of an alpha-olefin and another olefin or alpha-olefin (ethylene is defined to be an alpha-olefin for purposes of this invention). Preferably, the polymers are or include homo polyethylene, homo polypropylene, propylene copolymerized with ethylene and or butene, ethylene copolymerized with one or more of propylene, butene or hexene, and optional dienes. Preferred examples include thermoplastic polymers such as ultra low density polyethylene, very low density polyethylene ("VLDPE"), linear low density polyethylene ("LLDPE"), low density polyethylene ("LDPE"), medium density polyethylene ("MDPE"), high density polyethylene ("HDPE"), polypropylene, isotactic polypropylene, highly isotactic polypropylene, syndiotactic polypropylene, random copolymer of propylene and ethylene and/or butene and/or hexene, elastomers such as ethylene propylene rubber, ethylene

propylene diene monomer rubber, neoprene, and blends of thermoplastic polymers and elastomers, such as for example, thermoplastic elastomers and rubber toughened plastics.

[0049] As noted above, the particulates can be derived from other, non-polymer systems that produce, generate, or otherwise cause particulates to become charged, such as coal gasification, catalytic reforming, and cement production systems. As such, the particulates can also include, for example, coal, aluminosilicate catalyst, and cement.

[0050] Continuing with reference to Figure 1, in addition to the polymerization reactor 101 that can be in fluid communication with the one or more discharge vessels 155, the polymerization system 100 can further include one or more compressors 170 (only one is shown), and heat exchangers 175 (only one is shown). The polymerization system 100 can also include more than one reactor 101 arranged in series, parallel, or configured independent from the other reactors, each reactor having its own associated discharge tanks 155, compressors 170, and heat exchangers 175, or alternatively, sharing any one or more of the associated discharge tanks 155, compressors 170, and heat exchangers 175. For simplicity and ease of description, the polymerization system 100 will be further described in the context of a single reactor train.

[0051] The reactor 101 can include a cylindrical section 103, a transition section 105, and a velocity reduction zone or dome 107 ("top head"). The cylindrical section 103 is disposed adjacent the transition section 105. The transition section 105 can expand from a first diameter that corresponds to the diameter of the cylindrical section 103 to a larger diameter adjacent the dome 107. As mentioned above, the location or junction at which the cylindrical section 103 connects to the transition section 105 is referred to as the "neck" or the "reactor neck" 104. One or more cycle fluid lines 115 and vent lines 118 can be in fluid communication with the top head 107. The reactor 101 can include a fluidized bed 112 disposed within the cylindrical section 103 and in fluid communication with the top head 107.

[0052] In general, the height to diameter ratio of the cylindrical section 103 can vary in the range of from about 2:1 to about 5:1. The range, of course, can vary to larger or smaller ratios and depends, at least in part, upon the desired production capacity and/or reactor dimensions. The cross-sectional area of the dome 107 is typically within the range of from about 2 to about 3 multiplied by the cross-sectional area of the cylindrical section 103.

[0053] The velocity reduction zone or dome 107 has a larger inner diameter than the cylindrical section 103. As the name suggests, the velocity reduction zone 107 slows the velocity of the gas due to the increased cross-sectional area. This reduction in gas velocity allows particulates entrained in the upward moving gas to fall back into the bed, allowing primarily only gas to exit overhead of the reactor 101 through the cycle fluid line 115. The cycle fluid recovered via line 115 can contain less than about 10% wt, less than about 8% wt, less than about 5% wt, less than

about 4% wt, less than about 3% wt, less than about 2% wt, less than about 1% wt, less than about 0.5% wt, or less than about 0.2% wt of the particulates entrained in fluidized bed 112.

[0054] Suitable gas phase polymerization processes for producing the polymer product are described in U.S. Patent Nos. 3,709,853; 4,003,712; 4,011,382; 4,302,566; 4,543,399; 4,588,790; 4,882,400; 5,028,670; 5,352,749; 5,405,922; 5,541,270; 5,627,242; 5,665,818; 5,677,375; 6,255,426; European Patent Nos. EP 0802202; EP 0794200; EP 0649992; EP 0634421. Other suitable polymerization processes that can be used to produce the polymer product via line 117 can include, but are not limited to, solution, slurry, and high pressure polymerization processes. Examples of solution or slurry polymerization processes are described in U.S. Patent Nos. 4,271,060; 4,613,484; 5,001,205; 5,236,998; and 5,589,555.

[0055] A reactor feed via line 110 can be introduced to the polymerization system 100 at any location or combination of locations. For example, the reactor feed via line 110 can be introduced to the cylindrical section 103, the transition section 105, the velocity reduction zone 107, to any point within the cycle fluid line 115, or any combination thereof. Preferably, the reactor feed 110 is introduced to the cycle fluid in line 115 before or after the heat exchanger 175. A catalyst feed via line 113 can be introduced to the polymerization system 100 at any point. Preferably the catalyst feed via line 113 is introduced to the fluidized bed 112 within the cylindrical section 103.

[0056] The reactor feed in line 110 can include any polymerizable hydrocarbon or combination of hydrocarbons. For example, the reactor feed can be any olefin monomer including substituted and unsubstituted alkenes having two to 12 carbon atoms, such as ethylene, propylene, 1-butene, 1-pentene, 1-hexene, 1-heptene, 1-octene, 4-methylpent-1-ene, 1-decene, 1-dodecene, 1-hexadecene, and the like. The reactor feed can also include non-hydrocarbon gas(es) such as nitrogen and/or hydrogen. The reactor feed can enter the reactor at multiple and different locations. For example, monomers can be introduced into the fluidized bed in various ways including direct injection through a nozzle (not shown) into the fluidized bed. The polymer product can thus be a homopolymer or a copolymer, including a terpolymer, having one or more other monomeric units. For example, a polyethylene product could include at least one or more other olefin(s) and/or comonomer(s).

[0057] The reactor feed in line 110 can also include the one or more modifying components such as one or more induced condensing agents or ICAs. Illustrative ICAs include, but are not limited to, propane, butane, isobutane, pentane, isopentane, hexane, isomers thereof, derivatives thereof, and combinations thereof. The ICAs can be introduced to provide a reactor feed to the reactor having an ICA concentration ranging from a low of about 1 mol%, about 5 mol%, or about 10 mol% to a high of about 25 mol%, about 35 mol%, or about 45 mol%. Typical

concentrations of the ICAs can range from about 14 mol%, about 16 mol%, or about 18 mol% to a high of about 20 mol%, about 22 mol%, or about 24 mol%. The reactor feed can include other non-reactive gases such as nitrogen and/or argon. Further details regarding ICAs are described in U.S. Patent Nos. 5,352,749; 5,405,922; 5,436,304; and 7,122,607; and WO Publication No. 2005/113615(A2). Condensing mode operation, such as disclosed in U.S. Patent Nos. 4,543,399 and 4,588,790 can also be used to assist in heat removal from the fluid bed polymerization reactor.

[0058] The catalyst feed in line 113 can include any catalyst or combination of catalysts. Illustrative catalysts can include, but are not limited to, Ziegler-Natta catalysts, chromium-based catalysts, metallocene catalysts and other single-site catalysts including Group 15-containing catalysts, bimetallic catalysts, and mixed catalysts. The catalyst can also include AlCl_3 , cobalt, iron, palladium, chromium/chromium oxide or "Phillips" catalysts. Any catalyst can be used alone or in combination with any other catalyst.

[0059] Suitable metallocene catalyst compounds can include, but are not limited to, metallocenes described in U.S. Patent Nos.: 7,179,876; 7,169,864; 7,157,531; 7,129,302; 6,995,109; 6,958,306; 6,884,748; 6,689,847; 5,026,798; 5,703,187; 5,747,406; 6,069,213; 7,244,795; 7,579,415; U.S. Patent Application Publication No. 2007/0055028; and WO Publications WO 97/22635; WO 00/699/22; WO 01/30860; WO 01/30861; WO 02/46246; WO 02/50088; WO 04/022230; WO 04/026921; and WO 06/019494.

[0060] The "Group 15-containing catalyst" may include Group 3 to Group 12 metal complexes, wherein the metal is 2 to 8 coordinate, the coordinating moiety or moieties including at least two Group 15 atoms, and up to four Group 15 atoms. For example, the Group 15-containing catalyst component can be a complex of a Group 4 metal and from one to four ligands such that the Group 4 metal is at least 2 coordinate, the coordinating moiety or moieties including at least two nitrogens. Representative Group 15-containing compounds are disclosed in WO Publication No. WO 99/01460; European Publication Nos. EP0893454A1; EP 0894005A1; U.S. Patent Nos. 5,318,935; 5,889,128; 6,333,389; and 6,271,325.

[0061] Illustrative Ziegler-Natta catalyst compounds are disclosed European Patent Nos. EP 0103120; EP 1102503; EP 0231102; EP 0703246; U.S. Patent Nos. RE 33,683; 4,115,639; 4,077,904; 4,302,565; 4,302,566; 4,482,687; 4,564,605; 4,721,763; 4,879,359; 4,960,741; 5,518,973; 5,525,678; 5,288,933; 5,290,745; 5,093,415; and 6,562,905; and U.S. Patent Application Publication No. 2008/0194780. Examples of such catalysts include those comprising Group 4, 5 or 6 transition metal oxides, alkoxides and halides, or oxides, alkoxides and halide compounds of titanium, zirconium or vanadium; optionally in combination with a

magnesium compound, internal and/or external electron donors (alcohols, ethers, siloxanes, etc.), aluminum or boron alkyl and alkyl halides, and inorganic oxide supports.

[0062] Suitable chromium catalysts can include di-substituted chromates, such as $\text{CrO}_2(\text{OR})_2$; where R is triphenylsilane or a tertiary polyalicyclic alkyl. The chromium catalyst system may further include CrO_3 , chromocene, silyl chromate, chromyl chloride (CrO_2Cl_2), chromium-2-ethyl-hexanoate, chromium acetylacetonate ($\text{Cr}(\text{AcAc})_3$), and the like. Other non-limiting examples of chromium catalysts can be as discussed and described in U.S. Patent No. 6,989,344.

[0063] The mixed catalyst can be a bimetallic catalyst composition or a multi-catalyst composition. As used herein, the terms "bimetallic catalyst composition" and "bimetallic catalyst" include any composition, mixture, or system that includes two or more different catalyst components, each having a different metal group. The terms "multi-catalyst composition" and "multi-catalyst" include any composition, mixture, or system that includes two or more different catalyst components regardless of the metals. Therefore, the terms "bimetallic catalyst composition," "bimetallic catalyst," "multi-catalyst composition," and "multi-catalyst" will be collectively referred to herein as a "mixed catalyst" unless specifically noted otherwise. In one example, the mixed catalyst includes at least one metallocene catalyst component and at least one non-metallocene component.

[0064] In some embodiments, an activator may be used with the catalyst compound. As used herein, the term "activator" refers to any compound or combination of compounds, supported or unsupported, which can activate a catalyst compound or component, such as by creating a cationic species of the catalyst component. Illustrative activators include, but are not limited to, aluminoxane (*e.g.*, methylaluminoxane "MAO"), modified aluminoxane (*e.g.*, modified methylaluminoxane "MMAO" and/or tetraisobutyldialuminoxane "TIBAO"), and alkylaluminum compounds, ionizing activators (neutral or ionic) such as tri (n-butyl)ammonium tetrakis(pentafluorophenyl)boron may be also be used, and combinations thereof.

[0065] The catalyst compositions can include a support material or carrier. As used herein, the terms "support" and "carrier" are used interchangeably and are any support material, including a porous support material, for example, talc, inorganic oxides, and inorganic chlorides. The catalyst component(s) and/or activator(s) can be deposited on, contacted with, vaporized with, bonded to, or incorporated within, adsorbed or absorbed in, or on, one or more supports or carriers. Other support materials can include resinous support materials such as polystyrene, functionalized or crosslinked organic supports, such as polystyrene divinyl benzene polyolefins or polymeric compounds, zeolites, clays, or any other organic or inorganic support material and the like, or mixtures thereof.

[0066] Suitable catalyst supports are described in, for example, U.S. Patent Nos.: 4,701,432, 4,808,561; 4,912,075; 4,925,821; 4,937,217; 5,008,228; 5,238,892; 5,240,894; 5,332,706; 5,346,925; 5,422,325; 5,466,649; 5,466,766; 5,468,702; 5,529,965; 5,554,704; 5,629,253; 5,639,835; 5,625,015; 5,643,847; 5,665,665; 5,698,487; 5,714,424; 5,723,400; 5,723,402; 5,731,261; 5,759,940; 5,767,032; 5,770,664; and 5,972,510; and WO Publication Nos. WO 95/32995; WO 95/14044; WO 96/06187; WO 97/02297; WO 99/47598; WO 99/48605; and WO 99/50311.

[0067] The cycle fluid via line 115 can be compressed in the compressor 170 and then passed through the heat exchanger 175 where heat can be exchanged between the cycle fluid and a heat transfer medium. For example, during normal operating conditions a cool or cold heat transfer medium via line 171 can be introduced to the heat exchanger 175 where heat can be transferred from the cycle fluid in line 115 to produce a heated heat transfer medium via line 177 and a cooled cycle fluid via line 115. In another example, during idling of the reactor 101 a warm or hot heat transfer medium via line 171 can be introduced to the heat exchanger 175 where heat can be transferred from the heat transfer medium to the cycle fluid in line 115 to produce a cooled heat transfer medium via line 177 and a heated cycle fluid via line 115. The terms "cool heat transfer medium" and "cold heat transfer medium" refer to a heat transfer medium having a temperature less than the fluidized bed 112 within the reactor 101. The terms "warm heat transfer medium" and "hot heat transfer medium" refer to a heat transfer medium having a temperature greater than the fluidized bed 112 within the reactor 101. The heat exchanger 175 can be used to cool the fluidized bed 112 or heat the fluidized bed 112 depending on the particular operating conditions of the polymerization system 100, *e.g.* start-up, normal operation, and shut down. Illustrative heat transfer mediums can include, but are not limited to, water, air, glycols, or the like. It is also possible to locate the compressor 170 downstream from the heat exchanger 175 or at an intermediate point between several heat exchangers 175.

[0068] After cooling, all or a portion of the cycle fluid via line 115 can be returned to the reactor 101. The cooled cycle fluid in line 115 can absorb the heat of reaction generated by the polymerization reaction. The heat exchanger 175 can be of any type of heat exchanger. Illustrative heat exchangers can include, but are not limited to, shell and tube, plate and frame, U-tube, and the like. For example, the heat exchanger 175 can be a shell and tube heat exchanger where the cycle fluid via line 115 can be introduced to the tube side and the heat transfer medium can be introduced to the shell side of the heat exchanger 175. If desired, two or more heat exchangers can be employed, in series, parallel, or a combination of series and parallel, to lower or increase the temperature of the cycle fluid in stages.

[0069] Preferably, the cycle gas via line 115 is returned to the reactor 101 and to the fluidized bed 112 through fluid distributor plate ("plate") 119. The plate 119 can prevent polymer particulates from settling out and agglomerating into a solid mass. The plate 219 can also prevent or reduce the accumulation of liquid accumulation at the bottom of the reactor 101. The plate 219 can also facilitate transitions between processes which contain liquid in the cycle stream 115 and those which do not and vice versa. Although not shown, the cycle gas via line 115 can be introduced into the reactor 101 through a deflector disposed or located intermediate an end of the reactor 101 and the distributor plate 119. Illustrative deflectors and distributor plates suitable for this purpose are described in U.S. Patent Nos. 4,877,587; 4,933,149; and 6,627,713.

[0070] The catalyst feed via line 113 can be introduced to the fluidized bed 112 within the reactor 101 through one or more injection nozzles (not shown) in fluid communication with line 113. The catalyst feed is preferably introduced as pre-formed particulates in one or more liquid carriers (*i.e.* a catalyst slurry). Suitable liquid carriers can include mineral oil and/or liquid or gaseous hydrocarbons including, but not limited to, propane, butane, isopentane, hexane, heptane octane, or mixtures thereof. A gas that is inert to the catalyst slurry such as, for example, nitrogen or argon can also be used to carry the catalyst slurry into the reactor 101. In one example, the catalyst can be a dry powder. In another example, the catalyst can be dissolved in a liquid carrier and introduced to the reactor 101 as a solution. The catalyst via line 113 can be introduced to the reactor 101 at a rate sufficient to maintain polymerization of the monomer(s) therein.

[0071] As discussed above, the fluid via line 166 separated from the polymer product recovered via line 117 from the reactor 101 can be recycled via line 161 to the reactor 101. The fluid can include unreacted monomer(s), hydrogen, ICA(s), and/or inerts. Although not shown, the separated fluid via line 161 can be introduced to the cycle fluid in line 115. Although not shown, the polymer product via line 168 can be introduced to a plurality of purge bins or separation units, in series, parallel, or a combination of series and parallel, to further separate gases and/or liquids from the product. The particular timing sequence of the valves 157, 159, 167, can be accomplished by use of conventional programmable controllers which are well known in the art.

[0072] A product discharge system which can be alternatively employed is that disclosed in U.S. Patent No. 4,621,952. Such a system employs at least one (parallel) pair of tanks comprising a settling tank and a transfer tank arranged in series and having the separated gas phase returned from the top of the settling tank to a point in the reactor near the top of the fluidized bed. Other suitable product discharge systems that can be employed are described in PCT Publications

WO2008/045173 and WO2008/045172. One or more static probes 180 can be in communication with the internal volume of either the settling tank or the transfer tank. In another example, one or more static probes 180 can be in communication with both the settling tank and the transfer tank.

[0073] The reactor 101 can be equipped with one or more vent lines 118 to allow venting the bed during start up, operation, and/or shut down. The reactor 101 can be free from the use of stirring and/or wall scraping. The cycle line 115 and the elements therein (compressor 170, heat exchanger 175) can be smooth surfaced and devoid of unnecessary obstructions so as not to impede the flow of cycle fluid or entrained particulates.

[0074] The conditions for polymerization vary depending upon the monomers, catalysts, catalyst systems, and equipment availability. The specific conditions are known or readily derivable by those skilled in the art. For example, the temperatures can be within the range of from about -10°C to about 140°C, often about 15°C to about 120°C, and more often about 70°C to about 110°C. Pressures can be within the range of from about 10 kPag to about 10,000 kPag, such as about 500 kPag to about 5,000 kPag, or about 1,000 kPag to about 2,200 kPag, for example. Additional details of polymerization can be found in U.S. Patent No. 6,627,713.

[0075] Various systems and/or methods can be used to monitor and/or control a degree or level of fouling within the reactor 101. For example, if the polymerization system 100 is operated in condensed mode, a common technique for monitoring the polymerization can include monitoring a stickiness control parameter such as a reduced melt initiation temperature or "dMIT" value, which can provide an estimate as to the degree of polymer stickiness within the reactor 101. Another method for monitoring polymerization can include estimating acoustic emissions within the reactor 101, which can also provide an estimate as to the degree of polymer stickiness within the reactor 101. Additional details of monitoring a stickiness control parameter are described in U.S. Patent Application Publication No. 2008/0065360 and PCT Publication No. WO2008/030313. Another method for monitoring polymerization can include estimating acoustic emissions within the reactor, which can also provide an estimate as to the degree of polymer stickiness within the reactor. Additional details of monitoring a polymerization reactor via acoustic emissions are described in U.S. Publication No. 2007/0060721.

[0076] Other illustrative techniques that can also be used to reduce or eliminate fouling and/or sheeting can include the introduction of finely divided particulate matter to prevent agglomeration, as described in U.S. Patent Nos. 4,994,534 and 5,200,477. In another example, one or more continuity additives can be introduced to the reactor 101. Introducing a continuity additive can include the addition of negative charge generating chemicals to balance positive voltages or the addition of positive charge generating chemicals to neutralize negative voltage

potentials such as described in U.S. Patent No. 4,803,251. Antistatic substances can also be added, either continuously or intermittently to prevent or neutralize electrostatic charge generation. The continuity additive and/or antistatic substances, if used, can be introduced with the feed via line 110, the catalyst via line 113, a separate inlet (not shown), or any combination thereof.

[0077] The continuity additive can interact with the particulates and other components in the fluidized bed. For example, the continuity additive can reduce or neutralize static charges related to frictional interaction of the catalyst and polymer particulates. The continuity additive can also react or complex with various charge-containing compounds that can be present or formed in the reactor. The continuity additive can also react or complex with oxygenates and other catalyst poisons. The continuity additive can also be referred to as a static control agent.

[0078] As used herein, the term "continuity additive" refers to a compound or composition that when introduced into a gas phase fluidized bed reactor can influence or drive the static charge (negatively, positively, or to zero) in the fluidized bed. The continuity additive or combination of continuity additives can depend, at least in part, on the nature of the static charge. The particular continuity additive or combination of continuity additives can depend, at least in part, on the particular polymer being produced within the polymerization reactor, the particular spray dried catalyst system or combination of catalyst systems being used, or a combination thereof. Suitable continuity additives and uses thereof can be as discussed and described in European Patent No. 0 229 368; U.S. Patent Nos. 5,283,278; 4,803,251; and 4,555,370; and WO Publication No. WO2009/023111; and WO01/44322.

[0079] If an undesired amount or degree of charge is estimated to be on the polymer product introduced via line 158 to the discharge vessel 155, one or more operational adjustments or steps can be taken to reduce the amount of charge on the polymer product within the reactor 101. For example, one or more continuity additives and/or antistatic substances can be introduced to the reactor 101 to reduce the level of static charge on the polymer product therein. In another example, a rate of catalyst and/or feed introduction can be adjusted or modified, *e.g.*, increased or decreased. In still another example, the reactor 101 can be idled for a period of time, *i.e.* polymerization can be stopped within the reactor 101, but fluids can continue to cycle therein and a non-reacting fluidized bed can be maintained within the reactor during idling. Illustrative idling techniques can include those discussed and described in U.S. Provisional Patent Application having Serial No. 60/305,623. In yet another example, the reactor 101 can be shut down or "killed." Other actions or adjustments that can be taken to reduce the amount of charge on the polymer product within the reactor 101 can also include, but are not limited to, introducing one or more anti-static agents, replacing a source of the catalyst introduced via line

113 to the reactor 101 with a different source of the catalyst, changing the type of catalyst introduced via line 113 to the reactor 101, adjust a concentration of any condensing agents, if used, within the reactor 101, transition the polymerization reactor 101 to produce a different polymer product, and the like. Any one or more operational adjustments or steps can be taken alone, in any combination, and/or in any order to reduce the amount of charge on the polymer product within the reactor 101.

[0080] Described herein is a method and system for measuring the static charge on resin as it is discharged from a polymerization reactor. The method takes advantage of the cyclic nature of the product discharge system to detect current that flows to (and from) the static probe in the discharge tank as the tank is alternately filled with resin and emptied. This method represents an improvement over conventional static probe designs, as the method measures the total accumulated charge on the resin. Thus, overcoming a significant limitation of convention static probes that measure only the local rate of current flow from the probe tip, rather than the actual charge on the fluid bed. Having an accurate measurement of the charge on the fluid bed allows for better ability to quantify the static level in the reactor and thereby an improved basis for controlling the static by adjusting the flow of continuity additive to the reactor or idling or stopping the polymerization reaction if necessary.

[0081] The method described herein can be used to regulate the flow of continuity additive into the reactor. For example, in response to the estimated charge (Q), the amount of continuity additive being introduced to the polymerization reactor can be increased or decreased to maintain the charge at or near zero, or less than 1 $\mu\text{C/kg}$ of polymer, or less than 0.5 $\mu\text{C/kg}$ of polymer.

Example

[0082] To provide a better understanding of the foregoing discussion, the following non-limiting examples are provided. Although the examples are directed to specific embodiments, they are not to be viewed as limiting the invention in any specific respect. All parts, proportions and percentages are by weight unless otherwise indicated.

[0083] Figure 2 depicts a graphical depiction showing a current signal measured from a static probe 180 and electrometer 185 in communication with the internal volume 160 of the discharge vessel 155 as a polymer product was introduced to and removed from the discharge vessel 155. The y-axis is the current detected by the probe tip 181 and measured by the electrometer 185 in nano-amps (nA) and the x-axis is the time (seconds relative to the start of the introduction of polymer product to the discharge vessel).

[0084] The polymer product in this Example was prepared by polymerizing ethylene and hexene using a silica supported bis(1-methyl-3-butylcyclopentadienyl)zirconium dichloride metallocene catalyst with a methylaluminoxane activator in a gas phase polymerization reactor 101. The reactor was operated at a temperature of about 190°F and a pressure of about 107 psig. The polymerization was carried out using pentane as an ICA, which was present in a concentration of about 15.8 mol%. The amount of the cycle fluid that was condensed was about 22.4 wt%. The molar ratio of hydrogen to ethylene was 2.8. The molar ratio of hexene to ethylene was 1.7. The superficial gas velocity of the cycle gas through the polymerization reactor was about 2.5 ft/s. The polymer product produced within the reactor had a density of 0.9176 g/cm³, a bulk density of about 28.2 lb/ft³, and a melt index (I₂) of about 0.96 g/10 min. The polymer product was produced at a rate of about 125,000 lb/hr.

[0085] The flow control device 157 was opened at time zero and remained open for about 35 seconds as the polymer product via line 158 was introduced to the internal volume 160 of the discharge vessel 155, at which time the flow control device 157 was closed. The time period the flow control device 157 remained in the open position is indicated by reference numeral 205. The total mass of the polymer product introduced via line 158 to the discharge vessel 155 during the 35 seconds was about 2,100 kg. As shown in Figure 2, noise was observed in addition to the current or "displacement current" detected and measured via the probe tip 181 and electrometer 185. As discussed above, the noise is believed to be caused, at least in part, by contact between the polymer particulates and the probe tip 181.

[0086] The pressure within the discharge vessel 155 was about 2,200 kPa after the polymer product via line 158 was introduced thereto. Prior to opening the flow control device 167 and removing the polymer product via line 165 therefrom, fluid was removed via line 166 by opening the flow control device 159. The fluid removed via line 159 from the discharge vessel 155 reduced the pressure therein to about 1,100 kPa.

[0087] The flow control device 167 was opened at about 40 seconds (after the start of the introduction of the polymer product to the discharge vessel). The flow control device remained open for about 80 seconds during which the polymer product via line 165 was removed from the discharge vessel 155 and the flow control device 167 was then closed. The time period the flow control device 167 was in the open position is indicated by reference numeral 210. Closing the flow control device 167 corresponds to about 120 seconds (after the start of the introduction of the polymer product to the discharge vessel).

[0088] The current or "rebound current" detected via the probe tip 181 and measured via the electrometer 185 has much less noise as compared to the current measured during introduction of the polymer product to the discharge vessel 155. As such, for this particular example it was

preferred to use the measured current acquired during removal of the polymer product via line 167. The measured current reaches a negative peak of approximately -1.5 nA before gradually returning to zero as the charged polymer product exits the discharge tank 155.

[0089] Figure 3 depicts a close-up view of the graphical depiction shown in Figure 2. The area above the negative rebound current, area 305, was integrated and used in Equation 2. The negative sign used in Equation 2 indicates that the electrical charge on the polymer product is positive.

[0090] The value for the proportionality constant (κ) was determined according to the Computation method discussed and described above. For the particular discharge vessel used in this Example, the proportionality constant (κ) had a value of 68,000.

[0091] The integrated rebound current shown in Figure 3 was calculated to be approximately -29 nA-seconds, or -0.029 μC . Multiplying -0.029 μC by the negative of the proportionality constant (κ) or $-0.14 \times -68,000$ provides an estimate of about +1,970 μC as the total charge on the polymer product within the discharge vessel 155. Dividing +1,970 μC by the total mass of the polymer product introduced to the discharge vessel 155 provides an estimate of the specific charge on the polymer product. For this particular Example, the mass of the polymer product introduced to the discharge vessel 155 was 2,200 kg. Using Equation 3 above, the specific charge was determined to be +1,970 $\mu\text{C}/2,100 \text{ kg}$ or about +0.94 $\mu\text{C}/\text{kg}$.

[0092] All numerical values are "about" or "approximately" the indicated value, and take into account experimental error and variations that would be expected by a person having ordinary skill in the art.

[0093] Various terms have been defined above. To the extent a term used in a claim is not defined above, it should be given the broadest definition persons in the pertinent art have given that term as reflected in at least one printed publication or issued patent. Furthermore, all patents, test procedures, and other documents cited in this application are fully incorporated by reference to the extent such disclosure is not inconsistent with this application and for all jurisdictions in which such incorporation is permitted.

[0094] While the foregoing is directed to embodiments of the present invention, other and further embodiments of the invention may be devised without departing from the basic scope thereof, and the scope thereof is determined by the claims that follow.

CLAIMS:

What is claimed is:

1. A method for estimating a static charge on particulates, comprising:
 - measuring an electrical signal generated during an introduction of particulates to a discharge vessel or an electrical signal generated during removal of the particulates from the discharge vessel;
 - integrating the measured electrical signal; and
 - estimating a total charge (Q) on the particulates introduced to the discharge vessel at least in part by using the integrated electrical signal.
2. A method for estimating a static charge on particulates, comprising:
 - introducing particulates from a polymerization reactor to an internal volume of a discharge vessel;
 - removing the particulates from the discharge vessel;
 - measuring a static probe electrical signal generated during the introduction of the particulates to the discharge vessel or a static probe electrical signal generated during the removal of the particulates from the discharge vessel;
 - integrating the measured electrical signal; and
 - multiplying the integrated electrical signal by a proportionality constant (κ) to provide an estimated charge (Q) on the particulates introduced to the discharge vessel.
3. The method according to claim 1 or 2, wherein the electrical signal is measured using an electrometer in communication with a static probe having a probe tip in communication with the discharge vessel.
4. The method according to any one of claims 1 to 3, further comprising estimating a specific charge (q) on the particulates introduced to the discharge vessel.
5. The method according to any of claims 1, 3, or 4, wherein the particulates comprise a polymer product recovered from a polymerization reactor.
6. The method according to any one of claims 1 to 5, wherein the particulates comprise a polymer product recovered from a gas phase polymerization reactor.
7. The method according to any of claims 1 to 6, wherein the measured electrical signal is a current or voltage.

8. The method according to claim 1, wherein determining the estimated charge (Q) on the particulates comprises multiplying the integrated electrical signal by a proportionality constant (κ) to provide the estimated charge (Q).
9. The method according to claim 2 or 8, wherein the proportionality constant (κ) is determined by dividing an internal surface area of the discharge vessel by a surface area of a static probe tip that is in communication with the internal volume.
10. The method according to claim 2 or 8, wherein the proportionality constant (κ) is determined by removing a portion of the particulates from the discharge vessel; introducing the portion of the particulates to an electric charge measuring apparatus to provide an estimated charge on the portion of the particulates; and dividing the estimated charge by the integrated electrical signal to provide the proportionality constant (κ).
11. The method according to claim 2 or 8, wherein the proportionality constant (κ) is derived by performing calculations to determine simulated electric fields within the discharge vessel and a resulting electrical signal generated during introduction of the particulates, a resulting electrical signal generated during removal of the particulates, or both, as a function of a level of particulates in the discharge vessel.
12. A method for polymerizing olefins comprising:
 - (a) polymerizing olefins in a polymerization reactor to form polymer particulates;
 - (b) removing the polymer particulates from the polymerization reactor and introducing the polymer particulates to a discharge vessel; and
 - (c) estimating the static charge on the polymers particulates according to the method of any one of claims 1 to 11.
13. The method according to claim 12, further comprising introducing one or more continuity additives to the polymerization reactor in response to the estimated charge (Q).
14. The method according to claim 12, further comprising idling the polymerization reactor or stopping the polymerization reactor in response to the estimated charge (Q).
15. A system for estimating an electrical charge on particulates, comprising:
 - a discharge vessel having an internal volume for receiving particulates;

a first conduit in fluid communication with the internal volume and adapted to introduce the particulates to the internal volume;

a second conduit in fluid communication with the discharge vessel and adapted to remove the particulates from the internal volume;

a static probe in communication with the internal volume and adapted to detect an electrical signal generated as the particulates are introduced to the internal volume, as the particulates are removed from the internal volume, or both; and

an electrometer in communication with the static probe and adapted to measure the electrical signal detected by the static probe.

16. The system according to claim 15, further comprising a polymerization reactor, wherein the first conduit introduces a polymer product produced in the polymerization reactor to the discharge vessel.
17. The system according to claim 15 or 16, wherein a probe tip of the static probe is disposed through a wall of the discharge vessel and extends into the internal volume.
18. The system according to any one of claims 15 to 17, further comprising a processor in communication with the electrometer, wherein the processor receives the measured electrical signal and estimates a charge (Q) on the particulates based on the measured electrical signal.
19. The system according to any one of claims 15 to 17, further comprising a processor in communication with the electrometer, wherein the processor receives the measured electrical signal to estimate a specific charge (q) on the particulates based on the measured electrical signal.
20. The system according to any one of claims 15 to 19, wherein the electrical signal measured by the electrometer is a current or a voltage.

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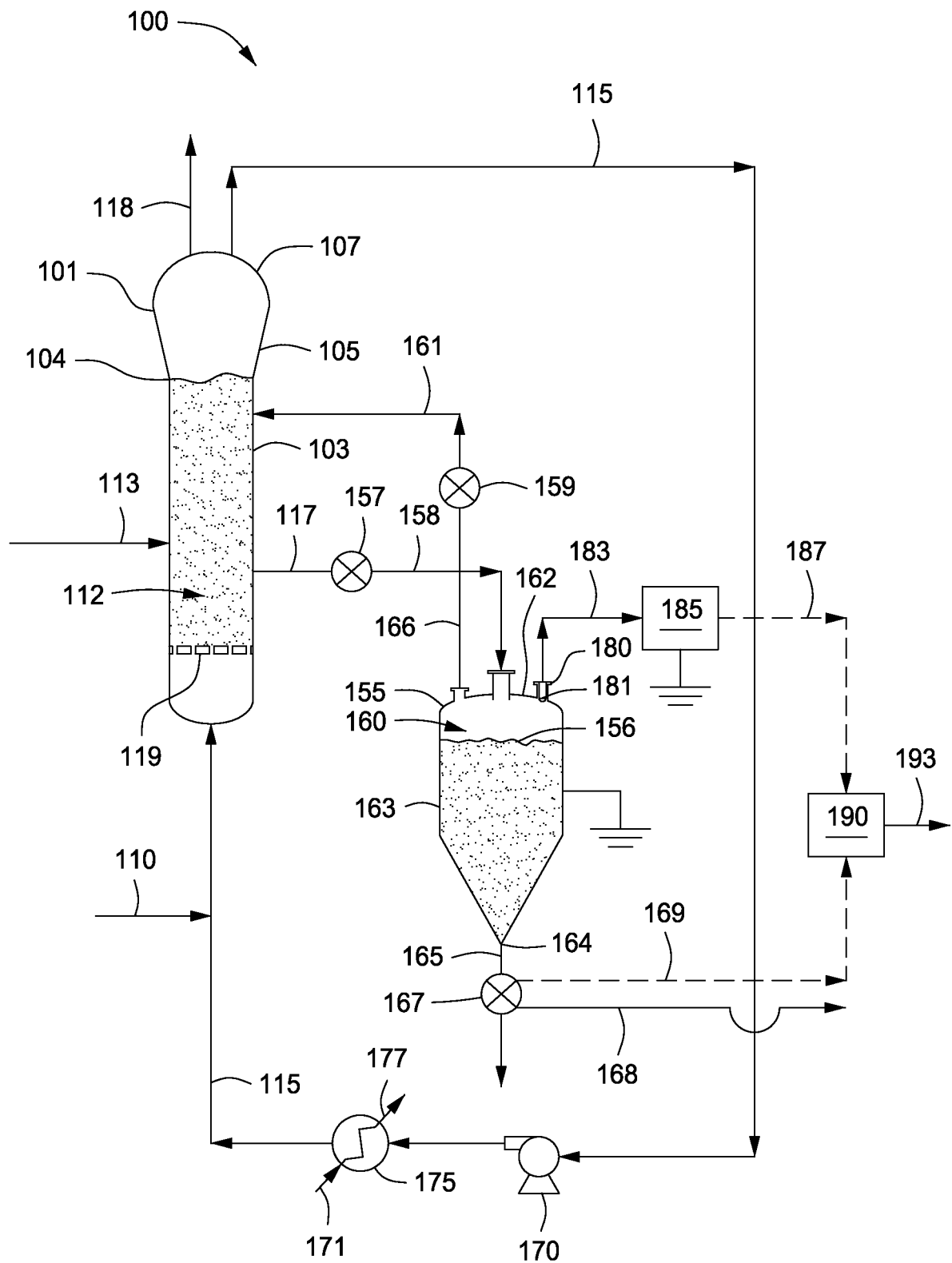


FIG. 1

FIG. 2

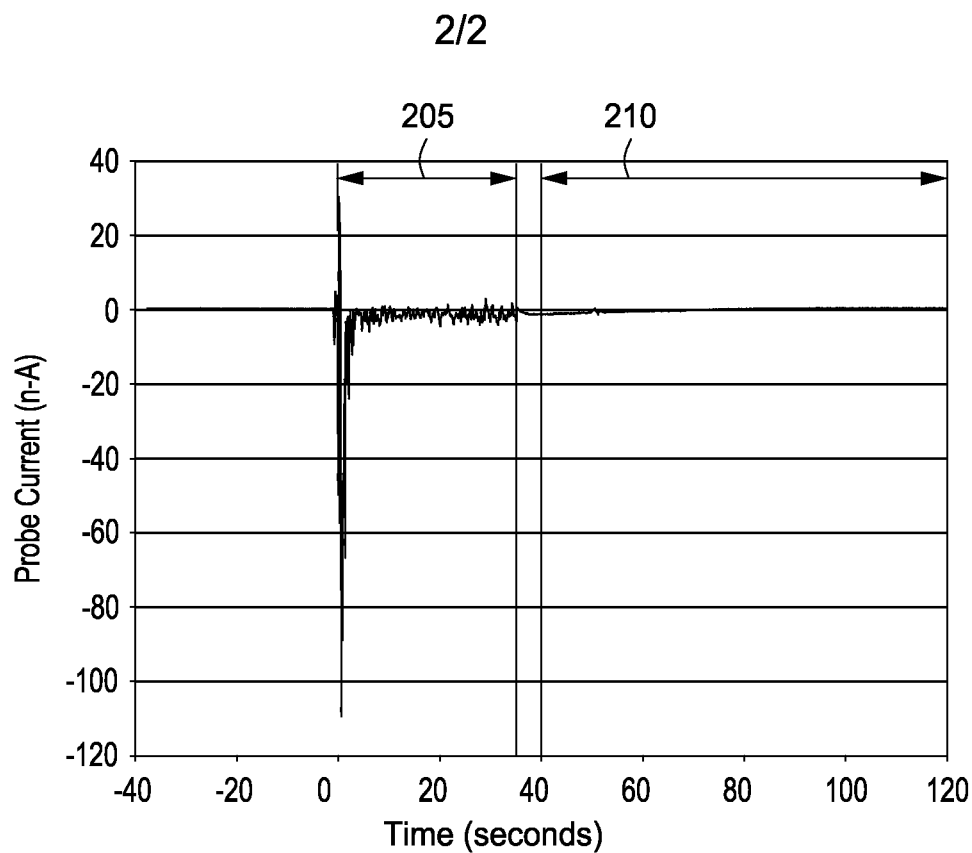
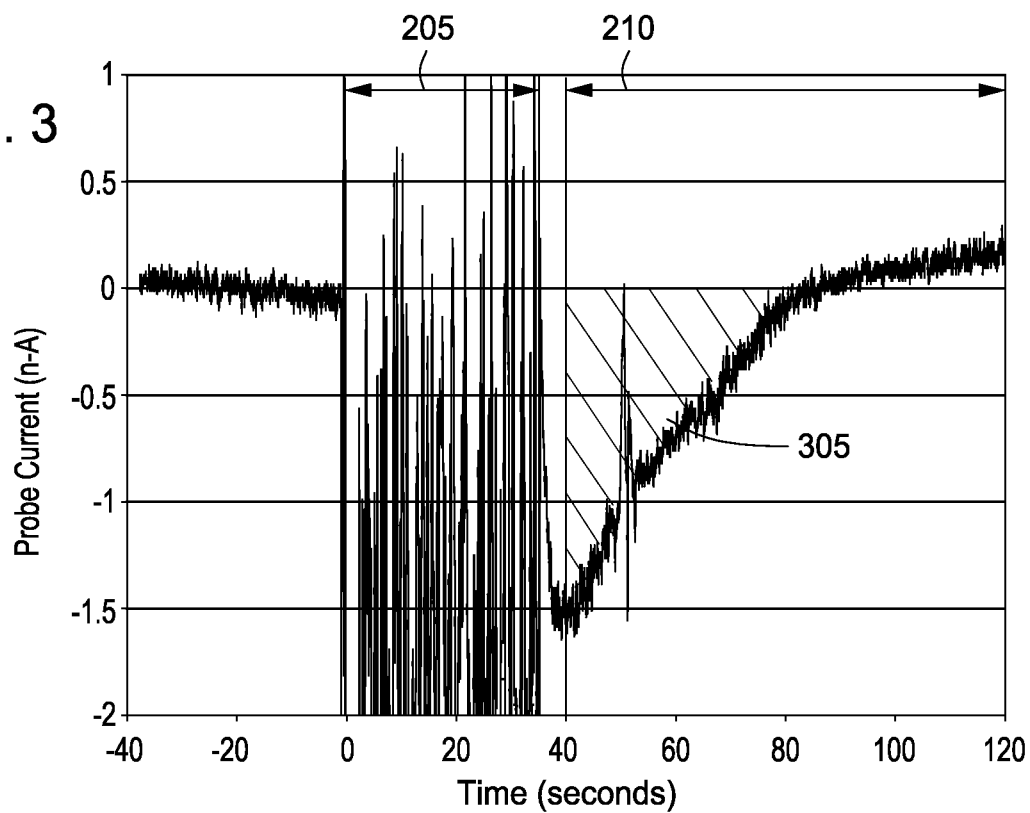


FIG. 3



INTERNATIONAL SEARCH REPORT

International application No
PCT/US2011/043327

A. CLASSIFICATION OF SUBJECT MATTER
INV. G01N27/60 G01N33/44
ADD.

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)
G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2003/133839 A1 (BARTILUCCI MARK PETER [US] ET AL) 17 July 2003 (2003-07-17)	1-8, 12-16, 18-20
Y	the whole document	9-11, 17
Y	WO 2008/016478 A2 (UNIVATION TECH LLC [US]; PANNEL RICHARD B [US]; MARKEL ERIC J [US]; HA) 7 February 2008 (2008-02-07) paragraph [0021]; figure 1	17
Y	GB 1 586 477 A (NAT RES DEV) 18 March 1981 (1981-03-18) page 3, line 1 - line 7	9-11
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☒ 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 document but published on or after the international filing date
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"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

4 October 2011

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INTERNATIONAL SEARCH REPORT

International application No

PCT/US2011/043327

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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International application No

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