

“sharper” and to have a more regular cutting surface than silicon carbide resulting in more of a descaling effect with diamond powder compared to debundling with silicon carbide.

**[0053]** As the tumbler **32** debundles/descals the CNTs, the CNTs are also separated from the catalyst seeds of the CNT bundles. These metallic catalyst seeds are attracted by the magnetic element **59** to the upstream end **32a** of the tumbler, and mostly remain in compartment **40b**. Bundled tubes and grit too large to pass through the first filter **50b** also largely remain in compartment **40b**. Typically a small percentage of bundled carbon nanotubes are small enough to pass through mesh **56** of the first filter **50** into the second cylindrical segment **42**, as do catalyst particles that are not magnetically bound to the magnetic element **59**. The debundled CNTs and amorphous coating are not magnetically attracted to magnetic element **59**, and consequently flow downstream through the mesh **56** of the first filter cartridge **50** into the second compartment **42b** of cylindrical segment **42**. Depending upon the respective pore sizes of the meshes **56** of the filters **50**, **52**, etc., the CNTs are separated into different compartments **42b**, **44b**, etc. based on size. It is preferred that downstream meshes have equal or smaller pore sizes than upstream meshes. According to one exemplary embodiment, three meshes with pore sizes of 50 micron, 5 micron, and 1 micron are used. In this embodiment, the metallic catalyst seeds and shearing particles, which are significantly denser than the CNTs and strongly attracted to the magnetic element **59** to the upstream end **32a** of the tumbler, mostly remain in compartment **40b** of the rotating tumbler **32**. The distribution of the lower density CNTs in the recirculated pumping system is restricted by the pore size and volume in each of the filtered zones. All CNTs with hydrodynamic sizes greater than the pore size of the intermediate filter will be retained in that zone. For example, a first 50 micron mesh filter restricts the bulk of the 120/220 grit [102/62  $\mu\text{m}$ ] silicon carbide grit and large CNT bundles. These larger CNT bundles do not pass through the first filter, but continued to be debundled and sheared until they have a smaller hydrodynamic size. The 5 micron filter traps the rest of the larger CNT aggregates, grit, etc. The third fraction <1 micron filtered contains the shortened CNTs as well as the finest amorphous grit residue. By adjusting the spacing between the filter rings approximately 80 to 90% of the closed volume of the system is partitioned for particles <1 micron. In this configuration, the smallest particles can recirculate but in practice are concentrated in compartment **44b**.

**[0054]** One advantage of this exemplary embodiment is that the rotating motion of the filter cartridges **50**, **52** is self-cleaning to prevent clogging at the meshes **56**. In the tumbler **32**, the filter meshes rotate as the pump (and/or gravitational forces) flowing liquid through the tumbler **32** applies a relatively low pressure against the upstream face of the filter meshes **56**. The applied low pressure serves to force a portion of the fluid and entrained particles through the mesh **56**. Viscous drag causes the entrained particles (including the CNTs) to move at a slower rotational speed than the rotating mesh **56**. Consequently, particles, even those smaller than the pore hydrodynamic radius, are in many instances rejected by the rotating mesh. Provision of the mesh with a relatively small thickness, 5 to 10 microns, allows those particles unable to make the rapid transition through the mesh to be washed back into the bulk fluid upstream from the filter, rather than clogging the filter. As discussed in greater detail below, chemically inert agitation spheres or particles may be placed

in one or more of compartments **40b**, **42b**, **44b** to collide with the mesh **56**, pulverizing or loosening small particles adherent to the mesh surface.

**[0055]** The tumbler **32** may be operated at a speed of, for example, 50 to 100 rpm, although the operating speed will vary depending upon the size of the tumbler **32**, the operating conditions, and the nature of the CNT starting material, the desired length of the resulting CNTs, and the dispersant concentration. In the case of SDS dispersant, the temperature of the medium inside the compartments **40b**, **42b**, **44b** may be maintained, for example, at or below about 10-12° C., such as at about 4° C. Although not limited, the temperature is preferably above 0° C. for SDS. Generally, the temperature should be above the freezing point of the aqueous-dispersant medium, but sufficiently low to increase viscosity.

**[0056]** The pump flow rate is preferably fairly low, but sufficient to reduce or prevent clogging. The pump flow rate may be, for example, about 50-100 ml/min for the micron-scale (Nylon) filters and 1-50 ml/min for nanoscale filters (PTCE), although the rate may deviate outside of this range depending on factors such as pore size and scale of operation. The flow rate will depend on the filter porosity (pore size), particle size and total recirculating system volume. A single experimental pump has a free flow rate in the 500-700 ml/min, but in practice once hooked up to nanoscale filters the rate drops to <1-10 ml/min. To increase flow rates several (2 or 3) pumps can be attached in series to achieve flow rates in the 1-50 ml/min range for the nanoscale filters (PTCE) and the 50-100 ml/min range for the microscale (Nylon) filters. As with the tumbler speed, the duration for which the tumbling is carried will vary depending on the properties of starting CNT material and shearing material. Those properties include the aggregation state of the CNTs (lower grade, heavily bundled CNTs) will take longer than high grade, better dispersed CNTs), and the hardness and diameter of the shearing particles. For example, in an embodiment using 120/220 silicon carbide grit and low grade CNT starting material, the initial tumbling time was set to 96 hours [range 12-120 hrs; @ 50 rpm 36,000 to 360,000 rotations]. In an embodiment using diamond powder 100,000 grit, CNTs processing times were reduced by approximately a factor of 10:10 hours, 30,000 rotations.

**[0057]** Depending on the source CNT material, the operation of tumbler **32** in terms of grit size, temperature, tumbler speed, pumping speed and processing duration can be altered to mitigate filter clogging that may arise due to unique characteristics, e.g., physical shape and size and/or chemical properties of the particles.

**[0058]** The pores of the filters **50**, **52** are not necessarily circular. As the sheared CNTs are released from their bundles, the pump assembly **36** induces a fluid flow in the chamber of tumbler **32** generally perpendicular to the plane in which the meshes of filter cassettes **50**, **52** extend. The fluid flow carries the debundled and shortened CNTs downstream towards first filter cassette **50** in the direction of the arrows **39** of FIG. 3. The first and second filter cassettes **50**, **52** constitute part of the tumbler **32** and are rotated by rotating system **34**. Accordingly, as the sheared CNTs approach the first filter **50**, the pores of the filter are in constant movement along a circumferential direction. To improve the likelihood of the axially traveling CNTs penetrating into and passing through the pores of the filters **50**, **52**, the pores may have an arcuate shape, extending lengthwise substantially circumferentially, as shown in FIG. 4B (not to scale).