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(54) **A MELT ATOMIZING NOZZLE AND PROCESS**

VERFAHREN UND DÜSE ZUM ATOMISIEREN VON SCHMELZE

PROCEDE ET TUYERE D'ATOMISATION D'UNE MASSE EN FUSION

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Description

The present invention relates to a high pressure gas atomizing nozzle for atomizing a melt, comprising; a melt supply member, a gas manifold for receiving high pressure gas, said manifold comprising a gas inlet in communication with a source of high pressure gas, a plurality of discrete gas discharge orifices disposed around the melt supply member and in communication with the manifold for discharging high pressure gas streams to atomise said melt.

Such a high pressure gas atomizing nozzle is known from DE-A-2 111 613. Such nozzles are used for atomizing a melt. The melt is supplied by the melt supply member and is atomized by the high pressure gas streams which are guided through the gas discharge orifices.

High pressure gas atomization (HPGA) as described in the Ayers and Anderson U. S. Patent 4,619,845 issued October 28, 1986, has shown considerable promise as a method of making very fine metal and alloy powder having a rapidly solidified microstructure with attendant control of alloyant segregation, grain size, supersaturation, and particle size, shape and distribution. The '845 patent describes atomization parameters required to effectively use the kinetic energy of supersonic (Mach 3 to 4) gas jet streams to disintegrate a melt into ultrafine, spherical powder particles. In particular, high pressure gas atomization in accordance with that patent employs an atomizing nozzle having multiple discrete, circumferentially spaced gas discharge orifices arranged about a nozzle melt supply tube having a central melt discharge orifice and adjacent frusto-conical surface. High pressure gas (typically an inert gas, such as argon, for reactive metals) is supplied to the gas discharge orifices for discharge in flush, laminar manner over the frusto-conical surface. The pressure of the gas supplied to the gas discharge orifices is selected at a high enough level (e.g., about $12,42 \cdot 10^6$ N/m^2 (1800 psig)) to establish a subambient pressure region immediately adjacent the melt discharge orifice to create an aspiration effect that draws melt out thereof for atomization at an apex region on the adjacent frusto-conical surface. The gas jet streams atomize the melt and form a narrow, supersonic spray containing very fine melt droplets that solidify rapidly as powder particles that are collected for further processing.

In addition to gas pressure, certain geometries/dimensions of the atomizing nozzle have been found to be important in achieving satisfactory atomization of the melt in the HPGA regime of operation. For example, the apex angle of the gas discharge orifices relative to the apex angle of the frusto-conical surface of the nozzle melt tube as well as the extension of the frusto-conical surface axially beyond the gas discharge orifices have been found to be important parameters that must be properly controlled and selected. Moreover, the orientation of the gas discharge orifices relative to the frusto-conical surface of the melt supply tube has also been found to be important. In particular, for optimum atomization, the gas discharge orifices should be flush with the frusto-conical surface and not offset therefrom. Detailed descriptions of the HPGA technique and the important parameters involved are set forth in the aforementioned patent and in the Anderson et al technical article (#1) "Fluid Flow Effects In Gas Atomization Processing", International Symposium on the Physical Chemistry of Powder Metals Production and Processing, TMS, Warrendale, PA. (1989) and the Figliola et al technical article (#2) "Flow Measurements In Gas Atomization Processes", Synthesis and Measurements in Gas Processing: Characterization and Diagnostics of Ceramic and Metal Particulate Processing, TMS, Warrendale, PA., pp. 39-47 (1989).

The second technical article referred to above describes modifications to certain geometries/dimensions of an original atomizing nozzle (developed by the Massachusetts Institute of Technology and described in its technical literature as an ultrasonic gas atomization nozzle) resulting in enhancement of the aspirating effect produced at the melt discharge orifice without affecting the location of the minimum pressure region relative thereto. In particular, the diameter of the melt supply tube was slightly enlarged to locate the gas discharge orifices flush with the frusto-conical surface adjacent the melt discharge orifice. An annular gas manifold was adopted although the gas jet ring diameter remained the same. The annular gas manifold was modified to have the gas discharge orifices communicate directly therewith rather via an intermediate annular manifold passageway as present in the M.I.T. device. The annular manifold was supplied with high pressure gas via a cylindrical conduit extending perpendicular to the manifold axis.

In attempting to atomize certain rare earth-transition metal magnetic alloys (e.g., rare earth-iron-boron alloys) using a gas atomizing nozzle modified as described in the aforementioned second technical article, the particular alloy chemistries involved were observed to adversely affect the atomization performance of the nozzle in so far as the particle sizes produced were distributed over a rather wide particle size range than expected, resulting in a higher than expected average particle size. In particular, a majority of the particle sizes produced were greater than an optimum particle size range (e.g., 3 to 44 micrometers, preferably 5 to 40 micrometers) where optimum magnetic properties are exhibited by the as-atomized particles for the particular alloy compositions involved. As a result, there is a need to further improve the performance of the gas atomizing nozzle in producing rare earth-transition metal magnetic alloy powder (e.g., rare earth-iron-boron alloy powder) to enable powder production with improved distribution of particle sizes in the optimum finer particle size range for the alloy compositions involved.

An object of the present invention is to provide a

high pressure gas atomizing nozzle and atomizing method characterized by improved atomization performance.

Another object on the present invention is to provide an improved, efficient high pressure gas atomizing nozzle and atomizing method capable of producing rapidly solidified powder particles, especially of rare earth-transition metal alloys, wherein the percentage (yield) of particles falling within a desired fine particle size range for optimum properties (e.g., magnetic properties for the rare earth-transition metal alloys) is substantially increased so as to thereby increase the yield of the atomizing process.

Still another object of the present invention is to provide an improved, efficient high pressure gas atomizing nozzle and atomizing method capable of producing fine, rapidly solidified powder particles at a lower gas pressure, thereby reducing the quantity and supply pressure of the gas required and the cost of producing the powder particles.

The objects according to the invention are achieved by a high pressure gas atomizing nozzle for atomizing a melt of the aforementioned kind, which is characterised by said manifold comprising a divergent first manifold chamber in communication with the gas inlet and a second manifold chamber in communication with said divergent manifold chamber and said gas discharge orifices.

This provides an improved high pressure gas atomizing nozzle of the type, wherein a plurality of discrete gas jets are so directed at a nozzle surface at elevated gas pressure as to establish an aspiration effect adjacent a melt discharge orifice and produce a supersonic spray containing atomized melt droplets. The improved atomizing nozzle includes a gas manifold having a divergent first manifold chamber (expansion chamber) between a gas inlet and second manifold chamber to minimize formation and maximize dissipation of expansion shock waves in the high pressure gas introduced from the inlet to the manifold. The first manifold chamber (expansion chamber), in effect, minimizes wall reflection shock waves as the high pressure gas enters the manifold to avoid formation of standing shock waves therein that hinder uniform filling of the manifold with high pressure gas. The uniformity and extent of filling of the gas manifold with the high pressure gas is thereby substantially improved to enhance atomization performance of the nozzle.

Optional features of the nozzle according to the invention are set out in dependent claims 2 - 13.

The second manifold chamber preferably comprises an arcuate (partially annular) manifold segment having an inner radius r_0 and outer radius r_1 relative to a central axis of the manifold. The divergent manifold chamber has a dimension r_2 relative to the same central axis such that $r_2 - r_0 \geq 2(r_1 - r_0)$, preferably $r_2 - r_0 \simeq 2.5 \ (r_1 - r_0)$.

The improved atomizing nozzle also includes improvements in the tangency of the gas jet discharge orifices to a nozzle central bore that receives a melt supply member (e.g., melt supply tube). The melt supply member includes a frusto-conical surface adjacent the melt discharge orifice. The improved tangency of the discharge orifices provides enhanced laminar flow of the gas jets or streams across the frusto-conical surface of the melt supply member to enhance the atomization performance of the nozzle. Moreover, the atomizing nozzle includes an increased number of gas discharge orifices disposed about a smaller diameter melt supply orifice to improve the uniformity of the gas curtain directed at the nozzle frusto-conical surface for atomization.

An improved two-piece melt supply member is provided for the atomizing nozzle to reduce melt freeze-up therein.

The present invention provides an improved atomizing nozzle and method for atomizing rare earth-transition metal alloys (e.g., rare earth-iron-boron) alloys into fine, rapidly solidified, generally spherical powder particles wherein the percentage (yield) of particles in the optimum size range for exhibiting magnetic properties in the as-atomized condition is substantially improved. For example, the weight percentage of particles having optimum magnetic properties is increased from about 25 weight % to greater than about 60 weight %, typically about 66-68 weight % of the atomized batch. Moreover, the improved atomization powder product is achieved at lower gas pressure as a result of the improved efficiency of the atomizing nozzle.

The mentioned nozzle provides an improved method (claim 14) of atomizing a rare earth-transition metal alloy melt by using a high pressure gas atomizing nozzle according to the invention and a method (claim 15) of making this high pressure gas atomizing nozzle.

The aforementioned objects and advantages of the present invention will become more readily apparent from the following detailed description taken in conjunction with the drawings.

Description of the Drawings

Figure 1 is a schematic view of an atomizing apparatus including the atomizing nozzle of the invention.

Figure 2 is a side elevation of an atomizing nozzle in accordance with one embodiment of the invention.

Figure 3 is a view of the atomizing nozzle along lines 3-3.

Figure 4 is a fragmentary sectional view of the atomizing nozzle showing gas jet discharge orifices aligned with the melt supply tube frusto-conical surface.

Figure 5 is a bottom elevation of the atomizing nozzle.

Figure 6 is a fragmentary sectional view of the nozzle melt supply tube after the gas discharge orifices are machined therein but before the final diameter of the central bore is machined.

Figure 7 is a bar graph illustrating the distribution in weight % of particles as a function of particle size (diam-

eter).

Figure 8 is a bar graph of illustrating the magnetic properties of as-atomized Nd-Fe-B alloy particles as a function of particle size.

Figure 9 is a similar bar graph for Nd-Fe-B-La particles.

Figure 10 is a bar graph for Nd-Fe-B alloy particles illustrating particle grain size as a function of particle size.

Detailed Description of the Invention

Referring to Figure 1, an atomization apparatus is shown for practicing one embodiment of the present invention where one or more environmentally protective layers are formed on the atomized particles in the apparatus in accordance with copending U.S. patent application "Environmentally Stable Reactive Alloy Powders And Method of Making Same" (attorney docket no. ISURF 1250). The present invention, however, is not limited to this embodiment wherein one or more protective layers are formed on the atomized powder particles in the atomization apparatus, and may be practiced to make uncoated as well as coated powder particles of various metals and alloys. The embodiment described herebelow wherein one or more protective layers are formed on the atomized powder particles is offered merely to illustrate one application of the invention in the production of powder particles. The improvements in atomization performance and particle size distributions described below by practicing the present invention are achievable irrespective of whether any protective layers are formed on the powder particles in the atomization apparatus.

The atomization apparatus includes a melting chamber 10, a drop tube 12 beneath the melting chamber, a powder collection chamber 14 and an exhaust cleaning system 16. The melting chamber 10 includes an induction melting furnace 18 and a vertically movable stopper rod 20 for controlling flow of melt from the furnace 18 to a melt atomizing nozzle 22 of the invention disposed between the furnace and the drop tube. The atomizing nozzle 22 is supplied with an inert atomizing gas (e.g., argon, helium) from a suitable source 24, such as a conventional bottle or cylinder of the appropriate gas. The atomizing nozzle 22 atomizes the melt in the form of a supersonic spray containing generally spherical, molten droplets D into the drop tube 12.

Both the melting chamber 10 and the drop tube 12 are connected to an evacuation device (e.g., vacuum pump) 30 via suitable ports 32 and conduits 33. Prior to melting and atomization of the melt, the melting chamber 10 and the drop tube 12 are evacuated to a level of 10^{-4} atmosphere to substantially remove ambient air. Then, the evacuation system is isolated from the chamber 10 and the drop tube 12 via the valves 34 shown and the chamber 10 and drop tube 12 are positively pressurized by an inert gas (e.g., argon to about 1.1 atmosphere) to prevent entry of ambient air thereafter.

The drop tube 12 includes a vertical drop tube section 12a and a lateral section 12b that communicates with the powder collection chamber 14. The drop tube vertical section 12a has a generally circular cross-section having a diameter in the range of 0,31-0,93 m (1 to 3 feet), a diameter of 0,31 m (1 foot) being used in the Examples set forth below. The diameter of the drop tube section 12a and the diameter of an optional reactive gas jet 40 are selected in relation to one another to provide a reactive gas zone or halo H extending substantially across the cross-section of the drop tube vertical section 12a at the zone H.

The length of the vertical drop tube section 12a is typically about 2,79 to about 4,96 m (9 to about 16 feet), a preferred length being 2,79 m (9 feet) being used in the Examples set forth below, although other lengths can be used in practicing the invention. A plurality of temperature sensing means 42 (shown schematically), such as radiometers or laser doppler velocimetry devices, may be spaced axially apart along the length of the vertical drop section 12a to measure the temperature of the atomized droplets D as they fall through the drop tube and cool in temperature.

The optional reactive gas jet 40 referred to above is disposed at location along the length of the vertical drop section 12a where the falling atomized droplets D have cooled to a reduced temperature (compared to the droplet melting temperature) at which the droplets have at least a solidified exterior surface thereon and at which the reactive gas in the zone H can react with one or more reactive alloying elements of the shell to form a protective barrier layer (reaction product layer comprising a refractory compound of the reactive alloying element) on the droplets whose depth of penetration into the droplets is controllably limited by the presence of the solidified surface as will be described below.

In particular, the jet 40 is supplied with reactive gas (e.g., nitrogen) from a suitable source 41, such as a conventional bottle or cylinder of appropriate gas, through a valve and discharges the reactive gas in a downward direction into the drop tube to establish the zone or halo H of reactive gas through which the droplets travel and come in contact for reaction in-situ therewith as they fall through the drop tube. The reactive gas is preferably discharged downwardly in the drop tube to minimize gas updrift in the drop tube 12. The flow patterns established in the drop tube by the atomization and falling of the droplets inherently oppose updrift of the reactive gas. As a result, a reactive gas zone or halo H having a more or less distinct upper boundary B and less distinct lower boundary extending to the collection chamber 14 is established in the drop tube section 12a downstream from the atomizing nozzle in Figure 1. As mentioned above, the diameter of the drop tube section 12a and the jet 40 are selected in relation to one another to establish a reactive gas zone or halo that extends laterally across the entire drop tube cross-section. This places the zone H in the path of the falling droplets D so that substantially all of the droplets travel

therethrough and contact the reactive gas.

The temperature of the droplets D as they reach the reactive gas zone H will be low enough to form at least a solidified exterior surface thereon and yet sufficiently high as to effect the desired reaction between the reactive gas and the reactive alloying element(s) of the droplet composition. The particular temperature at which the droplets have at least a solidified exterior shell will depend on the particular melt composition, the initial melt superheat temperature, the cooling rate in the drop tube, and the size of the droplets as well as other factors such as the "cleanliness" of the droplets, i.e., the concentration and potency of heterogeneous catalysts for droplet solidification.

The temperature of the droplets when they reach the reactive gas zone H will be low enough to form at least a solidified exterior skin or shell of a detectable, finite shell thickness; e.g., a shell thickness of at least about 0.5 micrometer. Even more preferably, the droplets are solidified from the exterior surface substantially to the droplet core (i.e., substantially through their diametral cross-section) when they reach the reactive gas zone H. As mentioned above, radiometers or laser doppler velocimetry devices, may be spaced axially apart along the length of the vertical drop section 12a to measure the temperature of the atomized droplets D as they fall through the drop tube and cool in temperature, thereby sensing or detecting when at least a solidified exterior shell of finite thickness has formed on the droplets. The formation of a finite solid shell on the droplets can also be readily determined using a physical sampling technique in conjunction with macroscopic and microscopic examination of the powder samples taken at different axial locations downstream from the atomizing nozzle in the drop tube 12.

Referring to Figure 1, prior to atomization, a thermally decomposable organic material can optionally be deposited on a splash member 12c disposed at the junction of the drop tube vertical section 12a and lateral section 12b to provide sufficient carbonaceous material in the drop tube sections 12a,12b below zone H as to form a carbon-bearing (e.g., graphite layer) on the hot droplets D after they pass through the reactive gas zone H. The organic material may comprise an organic cement to hold the splash member 12c in place in the drop tube 12. Alternately, the organic material may simply be deposited on the upper surface or lower surface of the splash member 12c. In any event, the material is heated during atomization to thermally decompose it and release gaseous carbonaceous material into the sections 12a,12b below zone H. An exemplary organic material for use comprises Duco® model cement that is applied in a uniform, close pattern to the bottom of the splash member 12c to fasten it to the elbow 12e. Also, the Duco cement is applied as a heavy bead along the exposed uppermost edge of the splash member 12c after the initial fastening to the elbow. The Duco cement is subjected during atomization of the melt to temperatures in excess of 500°C so that the cement thermally decomposes and acts as a source of gaseous carbonaceous material to be released into drop tube sections 12a,12b beneath the zone H. The extent of heating and thermal decomposition of the cement and, hence, the concentration of carbonaceous gas available for powder coating is controlled by the position of the splash member 12c, particularly the exposed upper most edge, relative to the initial melt splash impact region and the central zone of the spray pattern. To maximize the extent of heating and thermal decomposition, additional Duco cement can be laid down (deposited) as stripes on the upper surface of the splash member 12c.

Alternately, a second optional jet 50 can be disposed downstream of the first supplemental reactive gas jet 40. The second jet 50 is adapted to receive a carbonaceous material, such as methane, argon laced with paraffin oil and the like, from a suitable source (not shown) for discharge into the drop tube section 12a to form a graphitic carbon coating on the hot droplets D after they pass through the reactive gas zone H.

Powder collection is accomplished by separation of the powder particles/gas exhaust stream in the tornado centrifugal dust separator/collection chamber 14 by retention of separated powder particles in the valved powder-receiving container, Fig. 1.

In accordance with the invention, the atomizing nozzle 22 comprises a first annular nozzle body member 100 and second annular nozzle body member 102 welded together at W1,W2 to provide a nozzle body 104. As shown best in Fig. 2, a large diameter separation/chill plate 106 is disposed above the nozzle body 104. The plate 106 includes a plurality (e.g., 3) of circumferentially spaced screw receiving holes (not shown) to receive screws by which the plate 106 and nozzle body mounting plate 104a are positioned relative to the bottom of the induction melting furnace 18, Fig. 2. Mounting plate 104a is welded to the nozzle body 104.

The first and second nozzle body members 100,102 define therebetween a generally annular, vertically elongated gas manifold 110 that extends along the vertical, central axis L of the manifold 110. The gas manifold 110 receives high pressure gas (e.g., typically argon or helium) from the source 24, such as a conventional $41,4 \cdot 10^6$ N/m^2 (6000 psi) bottle or cylinder of appropriate gas, via a gas supply conduit 114 and a gas inlet 116 formed on the nozzle body 104. In particular, the gas supply conduit 114 extends from the gas source 24 to the gas inlet 116 where the conduit is welded in the gas inlet 116 as shown in Fig. 3 so as to provide a leakproof connection to avoid high pressure gas leakage.

Disposed between the gas inlet 116 and a constant cross-section, arcuate manifold segment 118 (second manifold chamber) is a divergent first expansion region or chamber 120 (first manifold chamber) that functions to minimize formation and maximize dissipation of expansion shock waves in the high pressure gas introduced from the gas inlet 116 to the manifold. The divergent first manifold chamber 120 is configured and

dimensioned to this end. In particular, the divergent chamber 120 comprises first and second diverging walls 122,124 that are machined in the nozzle body member 100 along the vertical length thereof. The walls 122,124 each diverge at an angle of between about 20 to about 45°, preferably about 32 degrees, relative to the central axis LL of the gas inlet 116 as shown best in Fig. 3. The first and second walls 122,124 thereby define an included angle A therebetween. Preferably, the included angle A is 64°. Moreover, the inner and outer radii r_0 and r_1 of the arcuate second manifold chamber 118 relative to the vertical, central axis L of the manifold 110 and the distance r_2 of the manifold inlet wall 120a relative to the axis L are selected in a particular relationship to this same end. Generally, r_0, r_1 and r_2 are selected in accordance with the relationship, $r_2 - r_0 \geq 2(r_1 - r_0)$. Preferably, r_0, r_1 and r_2 are selected in accordance with the relationship $r_2 - r_0 \approx 2.5(r_1 - r_2)$ to achieve optimum functioning of the expansion region or chamber 120; i.e., minimization of wall reflection shock waves as the high pressure gas enters the manifold to avoid formation of standing shock waves therein that hinder uniform filling of the manifold with high pressure gas. In this way, the uniformity and extent of filling of the gas manifold with the high pressure gas is thereby substantially improved to enhance atomization performance of the nozzle 22. The above-described relationships of r_0, r_1 and r_2 are based on a two-dimensional analysis of the high pressure gas flow into the gas manifold 110.

The high pressure gas flows from the gas manifold 110 through a plurality of gas jet discharge orifices 130 spaced circumferentially apart about a nozzle melt supply member (tube) 132 having a central melt discharge orifice 132a. The melt supply tube 132 is received in a central cylindrical bore 133 of the nozzle body 104. The gas discharge orifices 130 define an apex angle AA that preferably coincides with the apex angle of the frusto-conical surface 134 of the nozzle melt supply tube 132; namely, 45 degrees as shown in Fig. 4.

The melt supply member 132 preferably comprises an outer, tubular, metallic, (e.g., Type 304 stainless steel) member 132b, and an inner refractory tubular member 132c spaced apart by thermal insulating air space 132d to reduce the prospect of melt freeze-up in the melt supply member. The refractory tubular member 132c includes laterally extending, annular flange 132f disposed on an annular shoulder of the outer tubular member 132b. The outer, metallic tubular member 132b resists erosion at the surface 134. The refractory tubular member 132c may comprise boron nitride, machinable alumina, or graphite. The former (BN) is used in atomizing rare earth-transition metal alloys. The refractory tubular member 132c defines the melt discharge orifice 132a axially therethrough.

To further help in preventing melt freeze up, a cylindrical, tubular, metallic heat-reflector (not shown) or electrical heating device (not shown) can be disposed in the space 132d concentric with axis L.

In accordance with the present invention, the number of gas jet discharge orifices 130 is increased compared to the number used heretofore; e.g., as described in U.S. Patent 4,619,845 and the technical articles #1 and #2 referred to above in the Background of the Invention. For example, the number of discharge orifices 130 is increased from 18 to 20 while at the same time reducing the individual orifice diameter from 0,0787 to 0,0742 cm (0.0310 inch to 0.0292 inch) so as to maintain the same total discharge orifice exit area. In this way, the uniformity of the curtain of high pressure gas directed on the frusto-conical surface 134 of the nozzle melt feed tube 132 is enhanced.

In addition to increasing the number of gas jet discharge orifices 130, the present invention substantially improves the tangency T, Fig. 5, of the discharge orifices 130 relative to the central bore 133 of the nozzle body 104 to about 0,0051 cm (0.002 inch), preferably about 0,00254 cm (0.001 inch), in order to enhance laminar flow of the high pressure gas jets or streams over the frusto-conical surface 134. Laminar flow is known to be an important parameter in the atomization performance of the nozzle 22; e.g., see the aforementioned technical articles #1 and #2 referred to above. Improvement of the tangency T of the discharge orifices 130 to the central bore 133 allows orientation of the discharge orifices 130 in a more flush manner than has been possible with previous atomizing nozzles wherein the tangency of the discharge orifices 130 to the central bore 133 was typically in the range of 0,0102 to 0,013 cm (0.004 to 0.005 inch) and varied from one discharge orifice to another around the bore 133. The improved tangency of the discharge orifices 130 to the bore 133 in conjunction with a slight reduction in the size of the inner diameter of the melt supply discharge orifice 132a from 0,531 cm (0.209 inch) to preferably 0,475 cm (0.187 inch) contributes to improved atomization performance with respect to rare earth-transition metal alloys described below. The orifice 132a may have a diameter between about 0,531 cm (0.209 inch) to about 0,381 cm (0.150 inch).

In accordance with the invention, tangency of the discharge orifices 130 to the central bore 133 is substantially improved by first machining the bore 133 in the nozzle body member 102 to an initial, undersized lateral dimension (i.e., radius R') as shown in Fig. 6, then machining the discharge orifices 130 in the body member 102, and finally machining the central bore 133 to final, lateral dimension (i.e., radius R) by removing the envelope E of metal from the undersized initial bore. This particular machining sequence is carried out on a numerically controlled (NC) machine tool, such as an NC vertical milling machine available from Bridgeport.

The improved atomizing nozzle 22 described above is operable to atomize a melt in the manner described in U.S. Patent 4,619,845 and the aforementioned technical articles #1 and #2 by formation of a supersonic spray containing fine molten droplets, the teachings of which are incorporated herein by reference to this end.

Importantly, the improved atomizing nozzle 22 described above is capable of producing rapidly solidified powder particles, especially of rare earth-transition metal alloys, wherein the percentage (yield) of particles falling within a desired fine particle size range for optimum properties (e.g., magnetic properties for the rare earth-transition metal alloys) is substantially increased so as to thereby increase the yield of the atomizing process. The atomizing nozzle 22 of the invention is especially useful in atomizing rare earth-transition metal alloys that demonstrate desirable magnetic properties. The rare earth-transition metal alloys typically include, but are not limited to, Tb-Ni, Tb-Fe and other refrigerant magnetic alloys and rare earth-iron-boron alloys described in U.S. Patents 4,402,770; 4,533,408; 4,597,938 and 4,802931, the teachings of which are incorporated herein by reference, where the rare earth is selected from one or more of Nd, Pr, La, Tb, Dy, Sm, Ho, Ce, Eu, Gd, Er, Tm, Yb, Lu, Y and Sc. Lower weight lanthanides (Nd, Pr, La, Sm, Ce, Y, Sc) are preferred. Rare earth-iron-boron alloys, especially Nd-Fe-B alloys comprising about 26 to 36 weight % Nd, about 62 to 68 weight % Fe and about 0.8 to 1.6 weight % B, are particularly adapted for atomization in accordance with the invention.

Nd-Fe-B alloys rich in Nd (e.g., at least about 27 weight %) and rich in B (e.g., at least about 1.1 weight %) are preferred to promote formation of the hard magnetic phase, $Nd_2Fe_{14}B$, in an equiaxed, blocky microstructure, and minimize, preferably avoid, formation of the ferritic Fe phase in all particle sizes produced. The Nd-Fe-B alloys rich in Nd and B were found to be substantially free of primary ferritic phase when atomized in accordance with the invention, whereas the ferritic phase was observed in some particle sizes (e.g., 10 to 20 micrometers) for Fe rich and near-stoichiometric alloy compositions similarly atomized. Alloyants such as Co, Ga, La and others may be included in the alloy composition, such as 31.5 weight % Nd- 65.5 weight % Fe- 1.408 weight % B- 1.592 weight % La and 32.6 weight % Nd- 50.94 weight % Fe- 14.1 weight % Co- 1.22 weight % B- 1.05 weight % Ga.

With Nd-Fe-B type alloys, the Nd content of the alloy was observed to be decreased by about 1-2 weight % in the atomized powder compared to the melt as a result of melting and atomization, probably due to reaction of the Nd during melting with residual oxygen and formation of a moderate slag layer on the melt surface. The iron content of the powder increased relatively as a result while the boron content remained generally the same. The initial melt composition can be adjusted to accommodate these effects.

The following Examples are offered to further illustrate, but not limit, the present invention.

EXAMPLE 1

The melting furnace of Fig. 1 was charged with and Nd-Fe master alloy as prepared by thermite reduction,

an Fe-B alloy carbon-thermic processed and obtained from Shieldalloy Metallurgical Corp. and electrolytic iron from Glidden Co. The charge was melted in the induction furnace after the melting chamber and the drop tube were evacuated to 10^{-4} atmosphere and then pressurized with argon to 1.1 atmosphere to provide a melt of the composition 28 weight % Nd, 70.9 weight % Fe and 1.1 weight % B. The melt was heated to a temperature of 1650°C and supplied upon raising of the stopper rod to an atomizing nozzle of the prior art type used heretofore having an annular gas manifold fed high pressure argon via a suitable conduit. The gas manifold did not include a first manifold expansion chamber between the gas inlet and a second manifold chamber. Eighteen gas jet discharge orifices were present and defined an apex angle of 45 degrees that was equal to the apex angle of the frusto-conical surface of the nozzle melt supply tube. The discharge orifices were located as flush as possible to the frusto-conical surface given that they typically exhibited a tangency to the central bore of the nozzle body of about 0,0102 to 0,013 cm (0.004 to 0.005 inch) which varied from one orifice to another. The diameter of the melt discharge orifice 132a was 0,531 cm (0.209 inch).

Argon atomizing gas at $12,07 \cdot 10^6$ N/m² (1750 psig) was supplied to the atomizing nozzle. A reactive gas jet was located 190,5 cm (75 inches) downstream from the nozzle in the drop tube. Ultra high purity (99.995%) nitrogen gas was supplied to the jet at a pressure of 690000 N/m² (100 psig) for discharge into the drop tube to establish a nitrogen gas reaction zone or halo extending across the drop tube such that substantially all the atomized droplets traveled through the zone. At this location downstream from the nozzle, the droplets were determined to be at a temperature of about 1000°C or less, where at least a finite thickness solidified exterior shell was present thereon to control formation a protective reaction product layer on the particles in accordance with the teachings of copending U.S. patent application "Environmentally Stable Reactive Alloy Powders And Method Of Making Same" (attorney docket number ISURF 1250), the teachings of which are incorporated herein by reference.

After the atomized droplets traveled through the reaction zone, they were collected in the collection container of the collection chamber (e.g., see Fig. 1). The solidified powder product was removed from the collection chamber when the powder reached approximately 22°C. The powder collected was then size classified by sifting the powder through a full series of ASTM woven wire screens and by an automated size analysis technique based on laser light scattering by an ensemble of particles dispersed in a transparent fluid to determine the particle size distribution in the batch collected. The results of the size classification are shown in Fig. 7 under the designation BT-1-92 where it is evident that about 20 weight % of the particles were 125 micrometers in diameter, about 10 weight % were 75 micrometers in diameter, about 22.5 weight % were 63

micrometers in diameter, about 20 weight % were 45 micrometers in diameter and about 11 weight % were less than 38 micrometers in diameter. It is apparent that a majority of the particles are 63 micrometers and greater.

EXAMPLE 2

The melting furnace of Fig. 1 was charged in a similar manner to Example 1 with an Nd-16 weight % Fe master alloy as prepared by thermite reduction, an Fe-B alloy carbo-thermic processed and obtained from the Shieldalloy Metallurgical Corp. and electrolytic Fe obtained from Glidden Co. The charge was melted in the induction furnace after the melting chamber and the drop tube were evacuated to 10^{-4} atmosphere and then pressurized with argon to 1.1 atmosphere to provide a melt of the composition 31.51 weight % Nd, 65.49 weight % Fe, 1.408 weight % B, and 1.597 weight % La. The melt was heated to a temperature of 1650°C. After a hold period of 10 minutes to reduce (vaporize) Ca present in the melt (from the thermite reduced master alloy) to melt levels of 50-60 ppm by weight, the melt was fed to the atomizing nozzle 22 of the invention upon raising of the stopper rod.

The particular atomizing nozzle 22 used had an r_0 of 0,84 cm (0.3295 inch), r_1 of 1,16 cm (0.455 inch) and r_2 of 1,63 cm (0.642 inch). The diameter of the manifold wall (120a) was 0,95 cm (0.375 inch) with the inner diameter of the gas supply conduit (112) also being 0,95 cm (0.375 inch). The walls of the divergent manifold chamber defined a 64° included angle. The diameter of melt discharged orifice 132a was 0,47 cm (0.187 inch). In other respects, the atomizing nozzle used was as described above; e.g., 20 discharge orifices of 0,07 cm (0.0292) inch diameter defining a 45° apex angle with tangency of about 0,00254 cm (.001 inch) to the central bore 133.

Argon atomizing gas at $7,59 \cdot 10^6$ N/m² (1100 psig) was supplied to the atomizing nozzle 22. A reactive gas jet was located 190,50 cm (75 inches) downstream from the nozzle in the drop tube. Ultra high purity (99.995%) nitrogen gas was supplied to the jet at a pressure of 690000 N/m² (100 psig) for discharge into the drop tube to establish a nitrogen gas reaction zone or halo extending across the drop tube such that substantially all the atomized droplets traveled through the zone. At this location downstream from the nozzle, the droplets were determined to be at a temperature of about 1000°C or less, where at least a finite thickness solidified exterior shell was present thereon to control formation a protective reaction product layer on the particles.

After the atomized droplets traveled through the reaction zone, they were collected in the collection container of the collection chamber (e.g., see Fig. 1). The solidified powder product was removed from the collection chamber when the powder reached approximately 22°C. The powder collected was then size classified by passing the powder through a series of ASTM woven wire screens and by the automated size analysis to determine the particle size distribution in the batch collected. The results of the size classification are shown in Fig. 7 under the designation BT-1-190 where it is evident that about 2 weight % of the particles were 125 micrometers in diameter, about 2 weight % were 75 micrometers in diameter, about 4 weight % were 63 micrometers in diameter, about 18 weight % were 45 micrometers in diameter, and about 64-65 weight % were less than 38 micrometers in diameter. It is apparent that a majority of the particles are less than 38 micrometers in diameter.

EXAMPLE 3

A melt comprising 33.07 weight % Nd, 63.93 weight % Fe, 1.32 weight B and 1.68 weight % La was melted and atomized in a manner similar to that described in Example 2. The results of the size classification are shown in Fig. 7 under the designation BT-1-216 where it is evident that about 2 weight % of the particles were 125 micrometers in diameter about 3-4 weight % were 75 micrometers in diameter, about 3-4 weight % were 63 micrometers in diameter, about 10 weight % were 45 micrometers in diameter, and about 66-68 weight % were less than 38 micrometers in diameter. It is apparent that a majority of the particles are less than 38 micrometers in diameter.

The advantage of producing the alloy powder in the manner described wherein a majority of the particles are less than 38 micrometers in diameter is evident in Figs. 8 and 9. In these Figures, the magnetic properties (namely, coercivity, remanence and saturation) of as-atomized powder as a function of particle size is set forth for the aforementioned alloy BT-1-190 and another alloy BT-1-162 (32.5 weight % Nd-66.2 weight % Fe-1.32 weight % B). The alloy BT-1-162 was atomized in a manner similar to that of Examples 2-3. The Figures demonstrate that coercivity and, to a lesser extent, remanence appear to vary as a function of particle size in both alloys. Elevated levels of coercivity and remanence are observed in both alloys as particle size (diameter) is reduced below about 38 micrometers. On the other hand, saturation magnetization of both alloys remains relatively constant over the range of particle sizes. For alloy BT-1-162, the coercivity falls significantly as particle size is reduced below about 5 micrometers. These results correlate with grain size measurements which reveal a continuous decrease in grain size with reduced particle size; e.g., from a grain size of about 500 nm for 15-38 micrometers particles to about 40-70 for less than 5 micrometers particles; for example, as shown in Fig. 10 for alloy BT-1-162. Magnetic property differences between powder size classes were due to differences in the microcrystalline grain size within each particle class.

It is apparent that near optimum overall magnetic properties are exhibited by the as-atomized alloy particles in the general particle size (diameter) range of

about 3 to about 44 micrometers and, more particularly, about 5 to about 40 micrometers where the majority of the particles are produced by practice of the invention. Thus, the yield of as-atomized powder particles possessing useful magnetic properties is significantly enhanced in practicing the invention. Moreover, as is apparent from the Examples, these results are achieved at lower gas pressure.

Claims

1. A high pressure gas atomising nozzle for atomising a melt, comprising:

    a melt supply member (132),

    a gas manifold (110) for receiving high pressure gas, said manifold (110) comprising a gas inlet (116) in communication with a source of high pressure gas,

    a plurality of discrete gas discharge orifices (130) disposed around the melt supply member (132) and in communication with the manifold (110) for discharging high pressure gas streams to atomise said melt,

    characterised by
    said manifold (110) comprising a divergent first manifold chamber (120) in communication with the gas inlet (116) and a second manifold chamber (118) in communication with said divergent manifold chamber (120) and said gas discharge orifices (130).

2. The nozzle of claim 1, wherein said second manifold chamber (118) comprises a constant cross-section, arcuate manifold chamber.

3. The nozzle of claim 1, wherein the gas manifold (110) is disposed in a nozzle body (104) having a bore (133) that receives said melt supply member (132).

4. The nozzle of claim 3, wherein the gas discharge orifices (130) each intersect the bore (133) at a tangency (T) thereto not exceeding 0,0051 cm (.002 inches).

5. The nozzle of claim 4, wherein the tangency (T) of said discharge orifices (130) does not exceed 0,025 cm (.001 inches).

6. The nozzle of claim 1, wherein the arcuate manifold chamber (118) has an inner radius r_0 and an outer radius r_1 relative to a central axis of said manifold, and said expansion chamber has a dimension r_2 relative to said central axis wherein $r_2-r_0 \geq 2(r_1-r_0)$.

7. The nozzle of claim 6, wherein $r_2-r_0 \sim 2.5(r_1-r_0)$.

8. The nozzle of claim 1, wherein the expansion chamber (120) diverges at an included angle of about 40° to about 90°.

9. The nozzle of claim 8, wherein the included angle is about 64°.

10. The nozzle according to one of claims 1 to 9, wherein the melt supply member (132) comprises an inner refractory tubular member (132c) having a melt supply orifice (132a), and an outer metallic tubular member (132b) spaced from the inner refractory member (132c) to form a thermal insulating space between the inner and outer members.

11. The nozzle according to claim 10, wherein the inner member includes a lateral, annular flange (132f) disposed on an annular shoulder of the outer member (132b) proximate an upper end thereof.

12. The nozzle according to claim 10 or 11, wherein a heat reflector is disposed in the thermally insulating space between the inner and outer members.

13. The nozzle according to one of claims 10 to 12, wherein a heating device is disposed in the thermally insulating space between the inner and outer members.

14. A method of atomising a melt by using the high pressure gas atomising nozzle according to one of claims 1 to 13, **characterised by** supplying high pressure gas from the plurality of discrete gas discharge orifices (130) disposed around the melt supply member (132) and supplying the high pressure gas to the manifold (110) via the divergent first manifold chamber (120) disposed between the manifold gas inlet (116) and the second manifold chamber (118) so as to reduce standing shock wave patterns in said manifold (110) and thereby improve filling of said manifold with the high pressure gas that is discharged from the discharge orifices (130) for atomising the melt.

15. A method of making an improved high pressure gas atomising nozzle according to one of claims 1 to 13, having a bore for receiving a melt supply member (132), comprising the steps of:

    a) forming said bore (133) to an initial, undersized, lateral dimension,

    b) forming a plurality of discrete gas discharge orifices (130) about said bore (133) and intersecting therewith at a given angle, and

    c) forming the bore to a final, lateral dimension

larger than said undersized dimension such that the gas discharge orifices (130) are each at a tangency (T) to said final-dimensioned bore (133) not exceeding about 0,0051 cm (.002 inches).

Patentansprüche

1. Hochdruckgaszerstäubungsdüse zum Zerstäuben einer Schmelze mit:

   einem Schmelzenzuführbauteil (132),

   einem Gasverteiler (110) zum Aufnehmen des Hochdruckgases, wobei der Verteiler (110) einen Gaseinlaß (116) in Verbindung mit einer Quelle von Hochdruckgas aufweist,

   einer Vielzahl von einzelnen Gasauslaßöffnungen (130), die um das Schmelzenzuführbauteil (132) und in Verbindung mit dem Verteiler (110) zum Auslassen von Hochdruckgasströmen angeordnet sind, um die Schmelze zu zerstäuben,

   dadurch gekennzeichnet, daß
   der Verteiler (110) eine sich erweiternde erste Verteilerkammer (120) in Verbindung mit dem Gaseinlaß (116) aufweist, und eine zweite Verteilerkammer (118) in Verbindung mit der ersten sich erweiternden Verteilerkammer (120) und den Gasauslaßöffnungen (130) aufweist.

2. Düse nach Anspruch 1, bei der die zweite Verteilerkammer (118) eine bogenförmige Verteilerkammer mit konstantem Querschnitt aufweist.

3. Düse nach Anspruch 1, bei welcher der Gasverteiler (110) in einem Düsenkörper (104) angeordnet ist, der eine Bohrung (133) aufweist, die das Schmelzenzuführbauteil (132) aufnimmt.

4. Düse nach Anspruch 3, bei der die Gasauslaßöffnungen (130) jeweils um die Bohrung (133) mit einer Berührung (T) dazu schneiden, die 0,0051 cm (0,002 Inches) nicht übersteigt.

5. Düse nach Anspruch 4, bei der die Berührung (T) der Auslaßöffnungen (130) 0,025 cm (0,001 Inches) nicht übersteigt.

6. Düse nach Anspruch 1, bei der die bogenförmige Verteilerkammer (118) einen inneren Radius r_0 und einen äußeren Radius r_1 relativ zu einer mittigen Achse des Verteilers aufweist, und die Ausdehnungskammer eine Abmessung r_2 relativ zur mittigen Achse aufweist, wobei $r_2-r_0 \geq 2(r_1-r_0)$ ist.

7. Düse nach Anspruch 6, bei der $r_2-r_0 \approx 2,5(r_1-r_0)$ ist.

8. Düse nach Anspruch 1, bei der die Expansionskammer (120) sich mit einem eingeschlossenen Winkel von ca. 40 bis 90° erweitert.

9. Düse nach Anspruch 8, bei der der eingeschlossene Winkel ungefähr 64° ist.

10. Düse nach einem der Ansprüche 1 bis 9, bei der das Schmelzenzuführbauteil (132) ein inneres feuerfestes rohrförmiges Bauteil (132c) aufweist, welches eine Schmelzenzuführöffnung (132a) aufweist, und ein äußeres metallisches rohrförmiges Bauteil (132b) aufweist, welches von dem inneren feuerfesten Bauteil (132c) beabstandet ist, um einen thermisch isolierenden Raum zwischen den inneren und äußeren Bauteilen zu bilden.

11. Düse nach Anspruch 10, bei der das innere Bauteil einen seitlich verlaufenden ringförmigen Flansch (132f) aufweist, der auf einer ringförmigen Schulter des äußeren Bauteiles (132b) nahe einem oberen Ende davon angeordnet ist.

12. Düse nach Anspruch 10 oder 11, bei der ein Hitzereflektor in dem thermisch isolierenden Raum zwischen den inneren und äußeren Bauteilen angeordnet ist.

13. Düse nach einem der Ansprüche 10 bis 12, bei der eine Heizeinrichtung in dem thermisch isolierenden Raum zwischen den inneren und äußeren Bauteilen angeordnet ist.

14. Verfahren zum Zerstäuben einer Schmelze unter Verwendung einer Hochdruckgaszerstäubungsdüse gemäß einem der Ansprüche 1 bis 13, **dadurch gekennzeichnet**, daß Hochdruckgas durch die Vielzahl von diskreten Gasauslaßöffnungen (130) zugeführt wird, die um das Schmelzenzuführbauteil (132) angeordnet sind, und durch Zuführen des Hochdruckgases zu dem Verteiler (110) über die sich erweiternde erste Verteilerkammer (120), die zwischen dem Verteilergaseinlaß (116) und der zweiten Verteilerkammer (118) angeordnet ist, um stehende Stoßwellenmuster in dem Verteiler (110) zu reduzieren, und um dabei das Füllen des Verteilers mit dem Hochdruckgas zu verbessern, das durch die Auslaßöffnungen (130) ausgelassen wird zum Zerstäuben der Schmelze.

15. Verfahren zum Herstellen einer verbesserten Hochdruckgaszerstäubungsdüse gemäß einem der Ansprüche 1 bis 13, die eine Bohrung zum Aufnehmen eines Schmelzenzuführbauteiles (132) aufweist, mit den folgenden Schritten:

   a) Formen der Bohrung (133) mit einer anfänglichen zu kleinen Seitenabmessung,

b) Formen einer Vielzahl von diskreten Gas-auslaßöffnungen (130) um die Bohrung (133) und Schneiden damit unter einem gegebenen Winkel, und

c) Formen der Bohrungen auf eine endgültige seitliche Abmessung größer als die zu kleine Abmessung, so daß die Gasauslaßöffnungen (130) jeweils eine Berührung (T) zur endgültig dimensionierten Bohrung (133) aufweisen, die nicht größer ist als 0,0051 cm (0,002 Inches).

Revendications

1. Tuyère d'atomisation d'un gaz à haute pression pour atomiser une masse en fusion, comprenant :

   un élément d'amenée de masse en fusion (132),
   une tubulure à gaz (110) pour recevoir un gaz à haute pression, ladite tubulure (110) comprenant une entrée de gaz (116) en communication avec une source de gaz à haute pression, plusieurs orifices discrets de sortie de gaz (130) disposés autour de l'élément d'amenée de masse en fusion (132) et en communication avec la tubulure (110) pour appliquer des courants de gaz à haute pression et atomiser ladite masse en fusion,

   caractérisée en ce que
   ladite tubulure (110) comprend une première chambre de tubulure divergente (120) en communication avec l'entrée de gaz (116) et une seconde chambre de tubulure (118) en communication avec ladite chambre de tubulure divergente (120) et lesdits orifices de sortie de gaz (130).

2. Tuyère selon la revendication 1, dans laquelle ladite seconde chambre de tubulure (118) comprend une chambre de tubulure cintrée de section transversale constante.

3. Tuyère selon la revendication 1, dans laquelle la tubulure à gaz (110) est placée dans une structure de tuyère (104) ayant un alésage (133) qui reçoit ledit élément d'amenée de masse en fusion (132).

4. Tuyère selon la revendication 3, dans laquelle les orifices de sortie de gaz (130) coupent chacun l'alésage (133) avec un contact tangentiel (T) qui ne dépasse pas 0,0051 cm (0,002 pouce).

5. Tuyère selon la revendication 4, dans laquelle le contact tangentiel (T) desdits orifices de sortie (130) ne dépasse pas 0,025 cm (0,001 pouce).

6. Tuyère selon la revendication 1, dans laquelle la chambre de tubulure cintrée (118) a un rayon inté-rieur r_0 et un rayon extérieur r_1 par rapport à un axe central de ladite tubulure, ladite chambre d'expansion a une dimension r_2 par rapport audit axe central, avec $r_2 - r_0 \geq 2(r_1 - r_0)$.

7. Tuyère selon la revendication 6, dans laquelle $r_2 - r_0 \sim 2,5(r_1 - r_0)$.

8. Tuyère selon la revendication 1, dans laquelle la chambre d'expansion (120) diverge sous un angle inclus d'environ 40° à environ 90°.

9. Tuyère selon la revendication 8, dans laquelle l'angle inclus est d'environ 64°.

10. Tuyère selon l'une des revendications 1 à 9, dans laquelle l'élément d'amenée de la masse en fusion (132) comprend un élément tubulaire réfractaire intérieur (132c) ayant un orifice d'amenée de masse en fusion (132a), et un élément tubulaire métallique extérieur (132b) espacé de l'élément réfractaire intérieur (132c) pour former un espace d'isolation thermique entre les éléments intérieur et extérieur.

11. Tuyère selon la revendication 10, dans laquelle l'élément intérieur comprend une bride annulaire latérale (132f) placée sur un épaulement annulaire de l'élément extérieur (132b) à proximité d'une extrémité supérieure de ce dernier.

12. Tuyère selon la revendication 10 ou 11, dans laquelle un réflecteur de chaleur est placé dans l'espace d'isolation thermique entre les éléments intérieur et extérieur.

13. Tuyère selon l'une des revendications 10 à 12, dans laquelle un dispositif de chauffage est placé dans l'espace d'isolation thermique entre les éléments intérieur et extérieur.

14. Procédé d'atomisation d'une masse en fusion en utilisant la tuyère d'atomisation à gaz à haute pression selon l'une des revendications 1 à 13, caractérisé par l'application d'un gaz à haute pression venant des plusieurs orifices discrets de sortie de gaz (130) disposés autour de l'élément d'amenée de masse en fusion (132) et par l'application du gaz à haute pression à la tubulure (110) à travers la première chambre divergente de tubulure (120) placée entre l'entrée de gaz (116) de la tubulure et la seconde chambre de tubulure (118) afin de réduire les configurations d'ondes stationnaires de choc dans ladite tubulure (110) et par conséquent obtenir un meilleur remplissage de ladite tubulure par le gaz à haute pression qui sort des orifices de sortie (130) pour atomiser la masse en fusion.

15. Procédé de construction d'une tuyère d'atomisation

perfectionnée à gaz à haute pression selon l'une des revendications 1 à 13, ayant un alésage pour recevoir un élément d'amenée de masse en fusion (132), comprenant les étapes suivantes :

a) former ledit alésage (133) à une dimension latérale initiale sous-dimensionnée,
b) former une pluralité d'orifices discrets de sortie de gaz (130) autour dudit alésage (133) qui coupent ledit alésage sous un angle donné, et
c) former l'alésage à une dimension latérale finale supérieure à ladite dimension sous-dimensionnée de manière que les orifices de sortie de gaz (130) aient chacun un contact tangentiel (T) avec ledit alésage de dimension finale (133) qui ne dépasse pas environ 0,0051 cm (0,002 pouce).

Fig-1

Fig-2

Fig-3

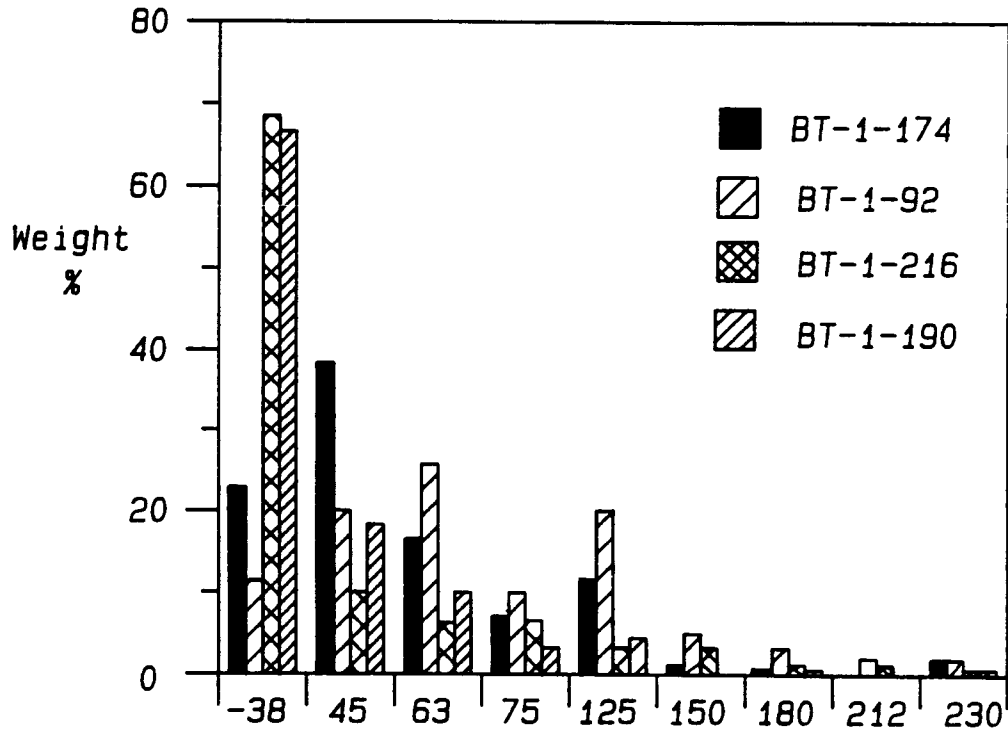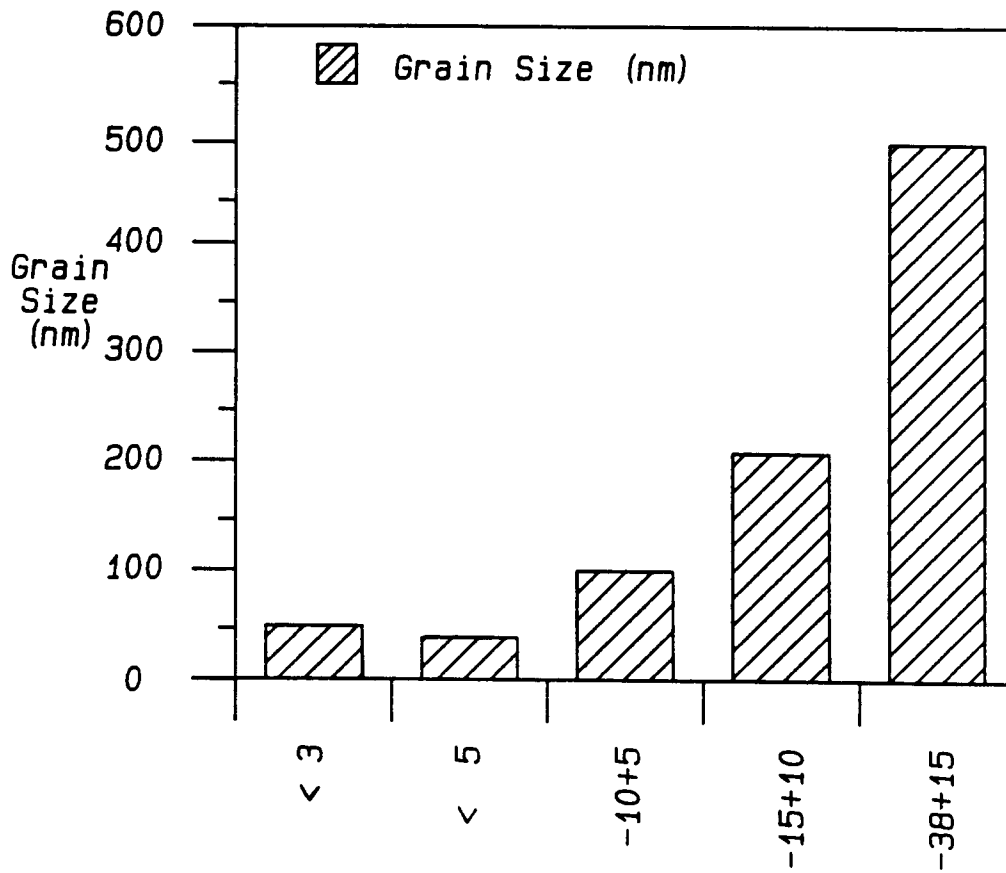$$r_2 - r_0 \cong 2.5(r_1 - r_0)$$

Fig-5

Fig-4

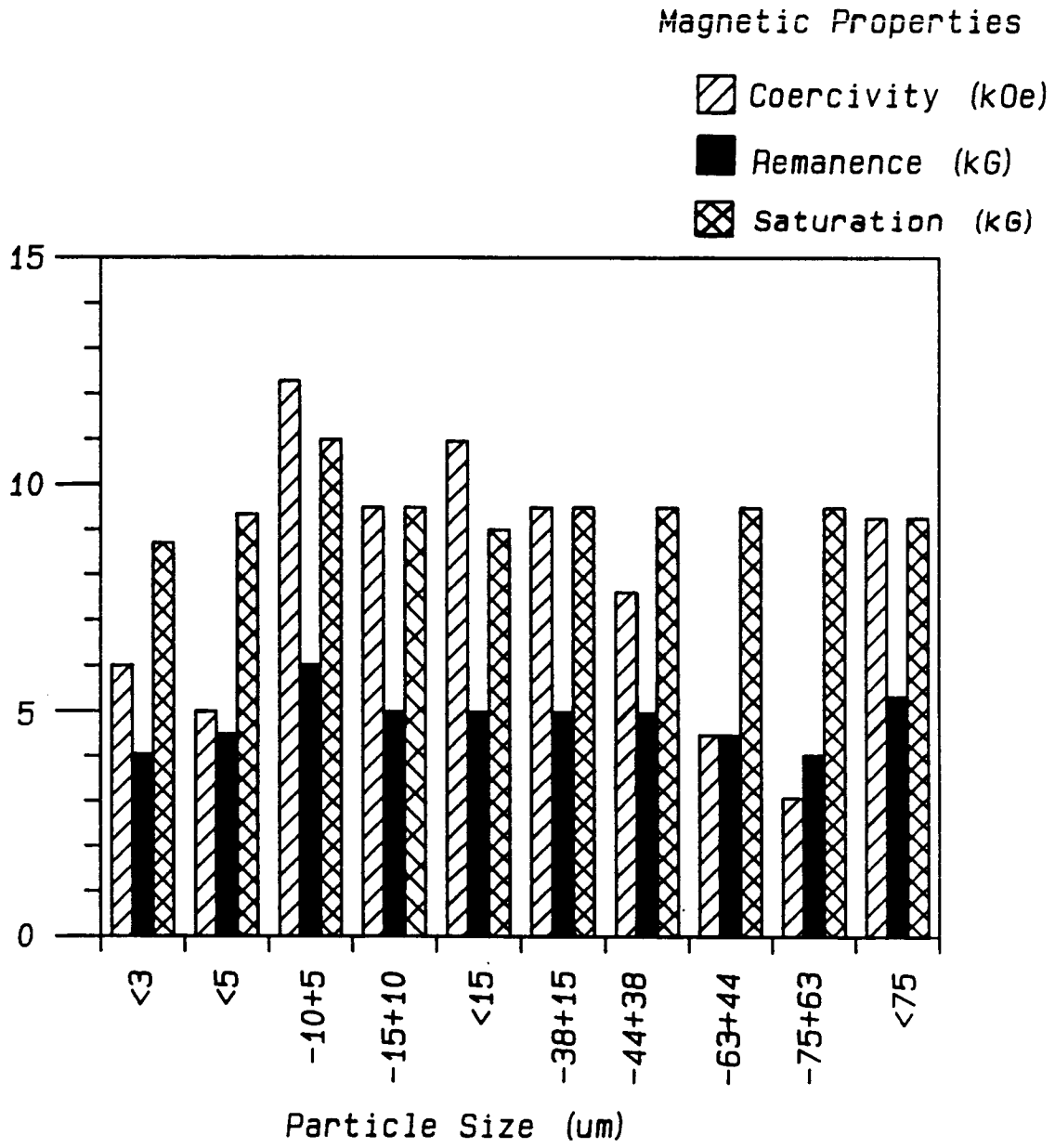*Fig-6*
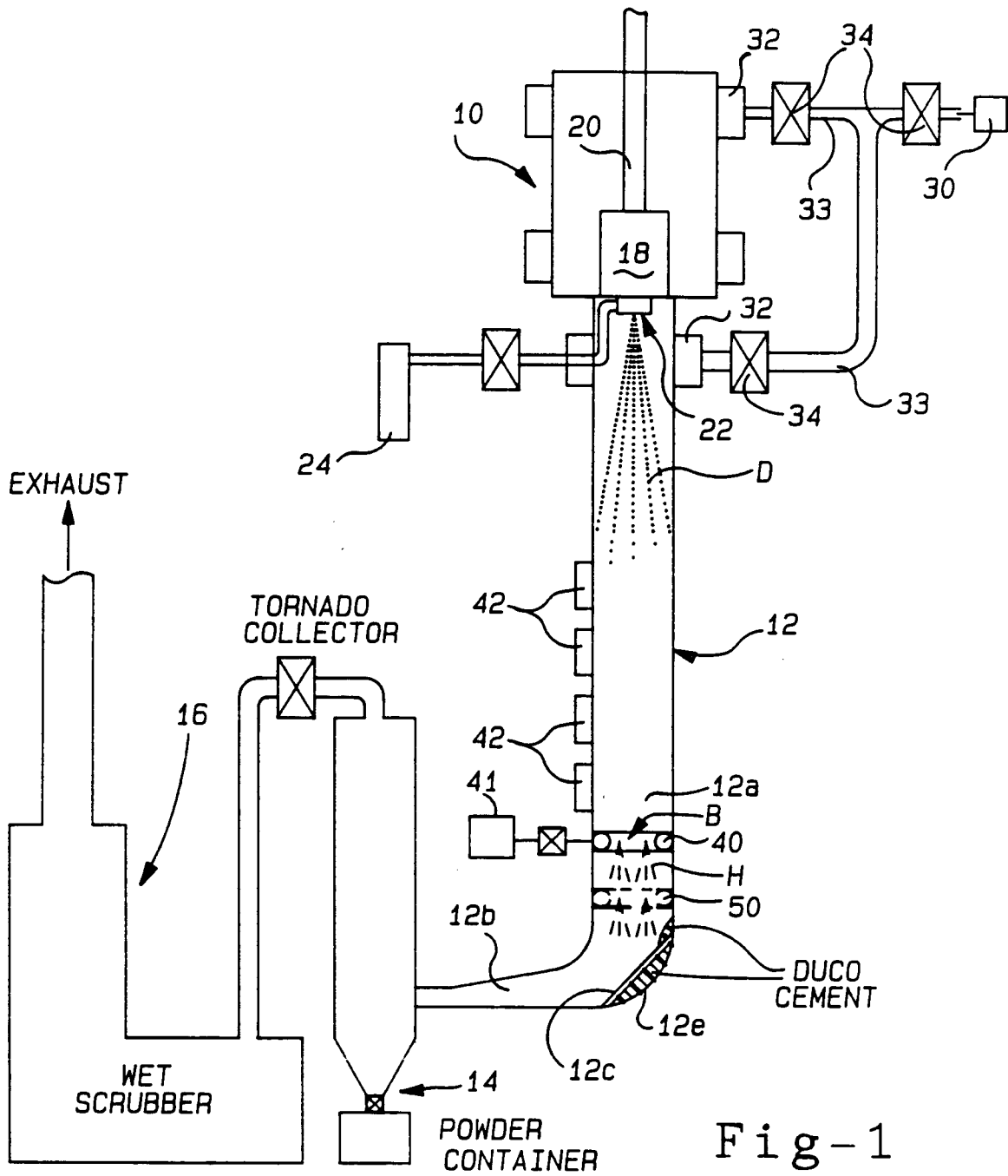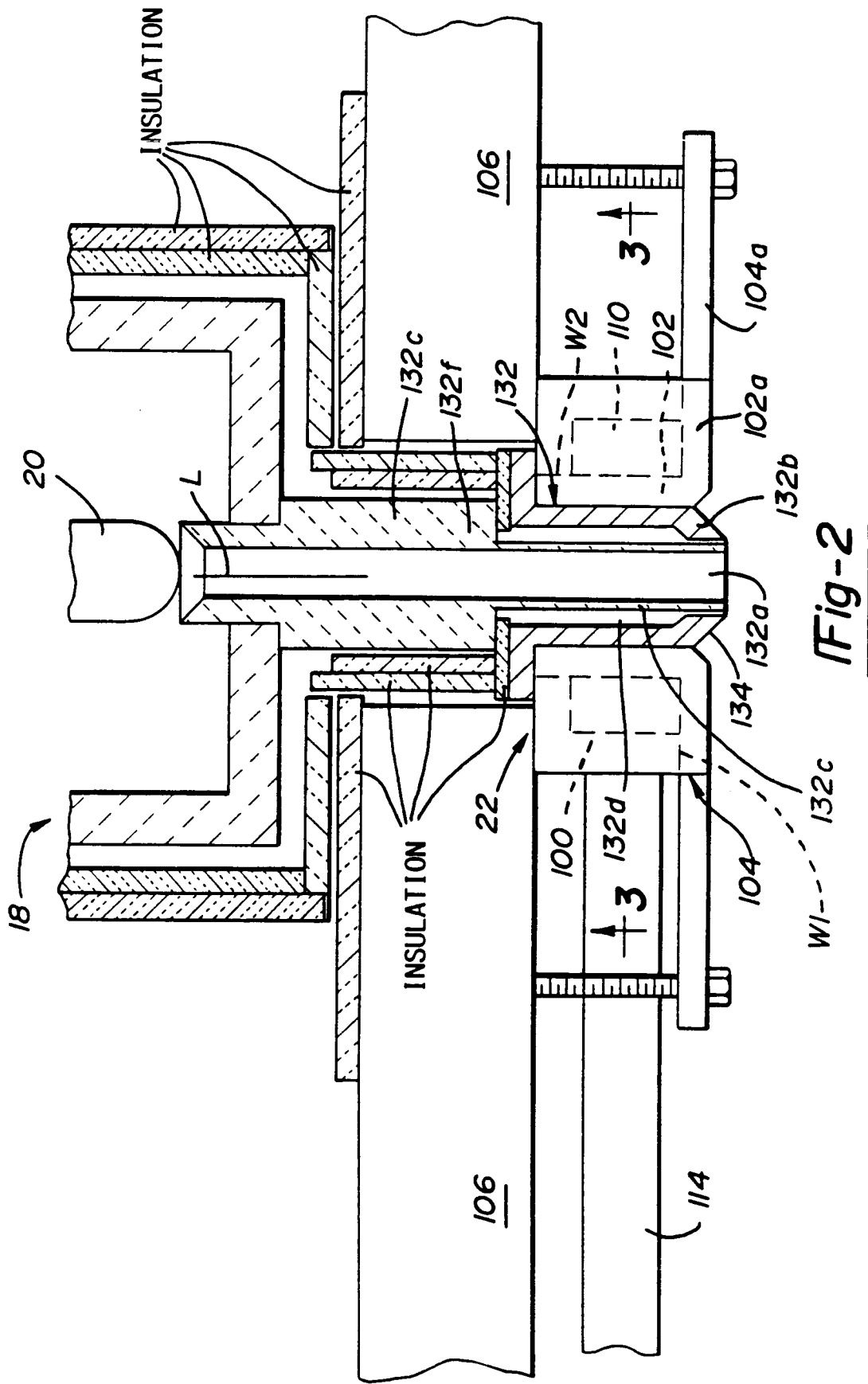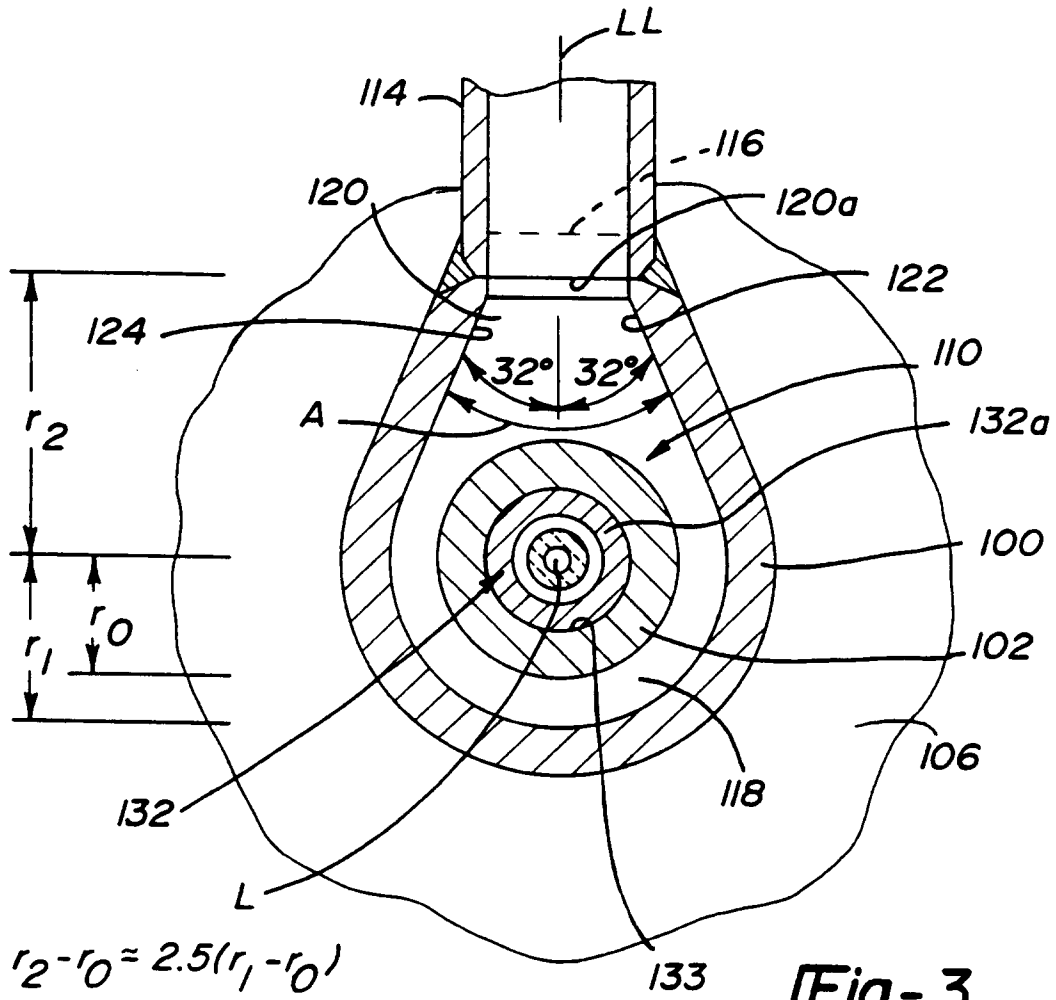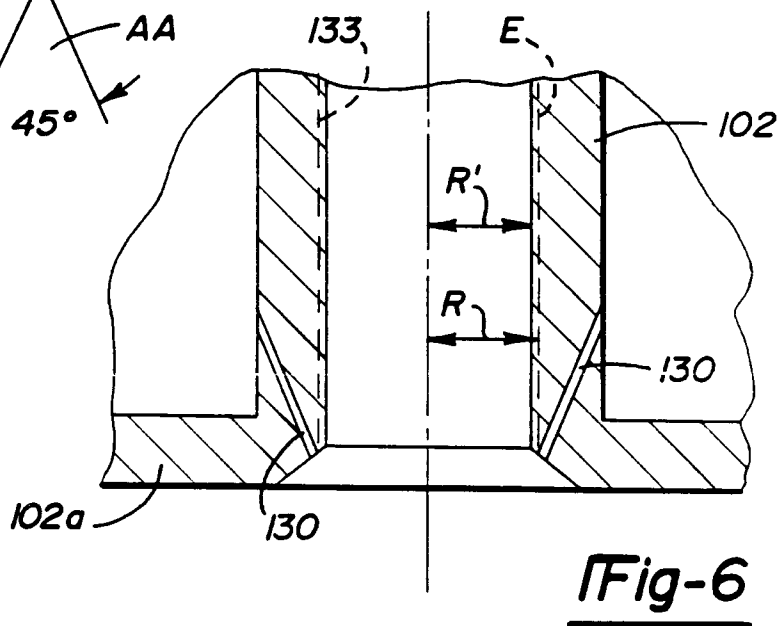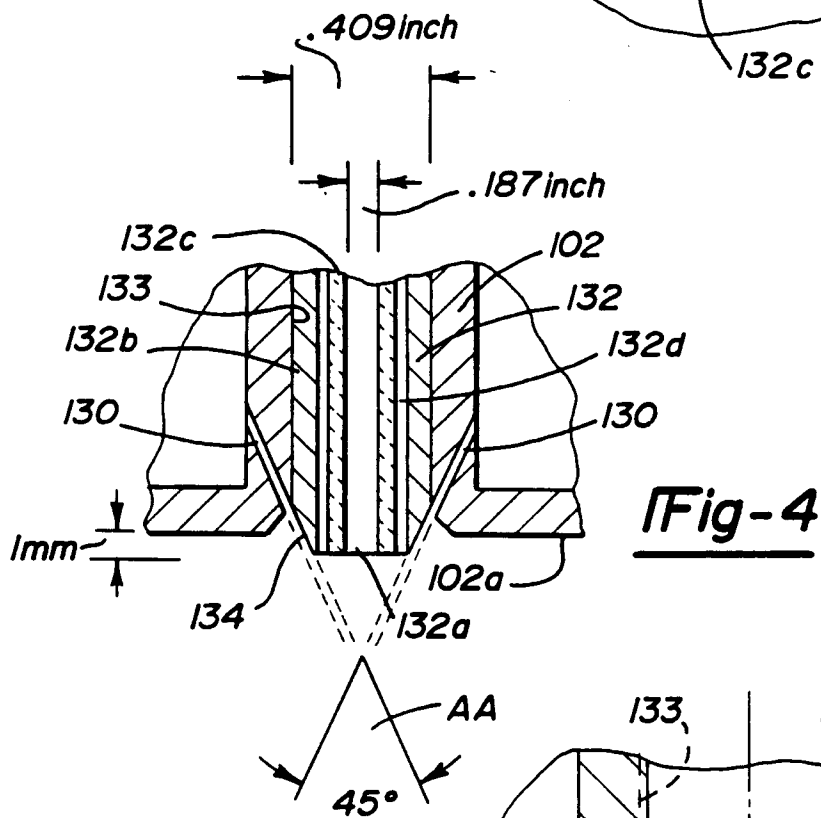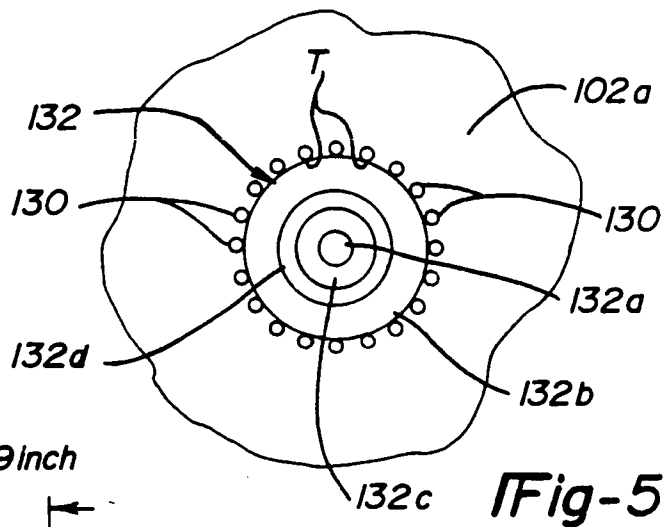
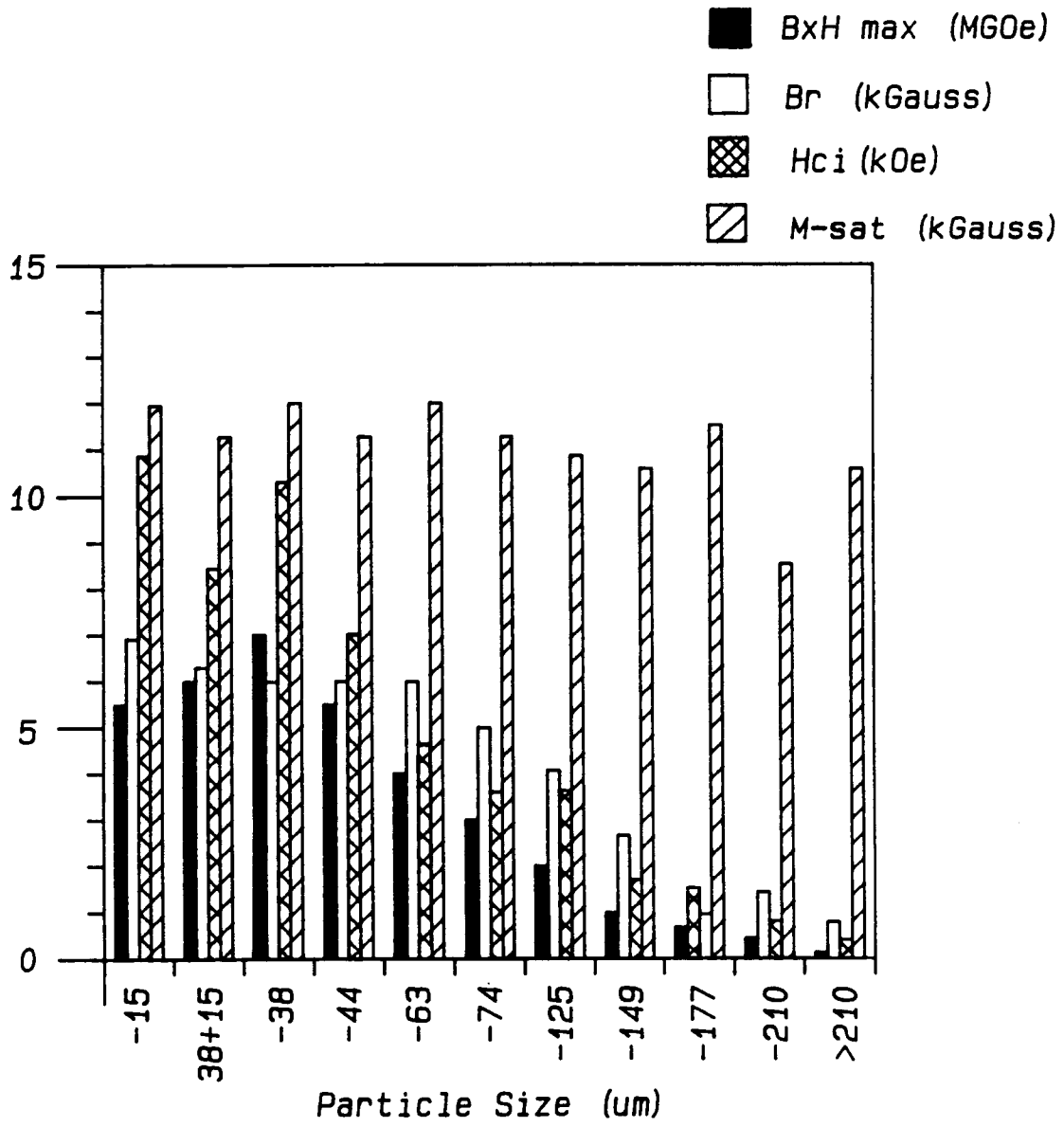Fig-7   Particle Size (um)

Fig-10   Particle Size (um)

Fig-8

Fig-9