Virtual Impact Fractionation of Superconductor Precursor Powders in Inert Gas Atmosphere

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Abstract—Virtual impactor fractionation has been used to remove all particles over a selectable micron-sized threshold in samples of precursor powders for MgB₂ and Nb₃Sn superconductors. In a virtual impactor the powder is dispersed in an aerosol stream and passed through a vane geometry in which particles less than a critical size follow the gas streamlines which turn abruptly into a collection chamber, while particles larger than the critical size pass undeflected into a reject chamber. The aerosol dispersion was made in an inert gas flow in order to prevent degradation of the powder by exposure to oxygen or moisture.

Index Terms—powder-in-tube, powder size, fine-filament, superconductor, composite wire

I. INTRODUCTION

The powder-in-tube (PIT) process is used to fabricate high-performance multi-filament superconducting wires of Nb₃Sn [1], Bi-2212 [2,3], and MgbB₂ [4,5]. While the specific powders, the synthesis routes, the sheath metal, and the PIT processing after powder filling are different in each case and indeed among the several practitioners of each superconductor, there is a common element that paces the ability to fabricate drawn multifilamentary composites of these materials. For high-energy physics applications the filament size is a critical parameter [6,7]. The PIT process entails cold-drawing the powder-filled tubes, restacking bundles of tubes, and repeated drawing and restacking to produce fine-filament round wire. The ultimate limit of this drawing and restacking comes when the tube apertures are reduced to ~twice the maximum particle size in the powder. Even if the frequency of such large particles is small, each occurrence of a break would result in rupture of the sheath and leakage of core material during subsequent heat treatment. Most of the precursor powders for the above superconductors are ground using either jet-milling or attritor milling. Either technique produces a roughly Gaussian particle size distribution, with large-size tails extending to ~4 times the mean particle size. That large-size tail is a predominant limit for producing fine-filament superconducting wire using the PIT process. Particles in the size range up to cannot be removed by simple mechanical sieving (the fines aggregate electrostatically and block the sieve mesh) and techniques utilizing cyclones cannot separate below ~10 μm size without passing large-size tails.

Virtual impactor sizing is the only technique that has the capability to provide near-zero-defect removal of particles above a chosen μm size threshold. The technique is illustrated in Figure 1. The powder is dispersed as an aerosol suspension in a buffer gas. The aerosol is passed through a vane geometry in which 10% of the flow goes straight while 90% of it is forced to make an abrupt right-angle bend. The fate of each particle is determined by its aerodynamic size. The principal parameter determining its operation is the Stokes number (Stk) given by

\[ Stk = \frac{\rho_p D^5 C U}{9 \mu w} \]

where \( \rho_p \) is particle density, \( D \) is the particle diameter, \( C \) is the slip correction factor, \( U \) is the throat velocity, \( \mu \) is the dynamic viscosity of the carrier fluid and w is the slot width. For \( Stk < 1 \) particles follow gas streamlines so that 90% go into the major flow; for \( Stk > 1 \) particles follow ballistic trajectories and so all go straight into the minor flow. The cutoff particle size \( D \) of a virtual impactor is defined by \( Stk = 1 \), when the collection is equally divided between the minor and the major flows.

![Figure 1. Particle separation in a Virtual Impactor](image)
II. DEVELOPMENT OF VI PROCESSING AT ATC

In an earlier phase of development [8,9] the apparatus shown in Figure 2 was used to evaluate VI separation of precursor powders for PIT superconductors. Excellent separation with a sharp separation threshold was achieved, as shown in Figure 3. It should be noted that the velocity profile in the inlet flow was flattened by the convergent vane geometry, so that the separation performance is the sharpest transition from major to minor flow transmission that has ever been recorded.

Four issues were identified that needed to be addressed in order to adapt the process for cost-effective manufacture of the precursor powders for superconductors:

- It is necessary to use inert buffer gas for the aerosol dispersion so that the powder is not exposed to oxygen, water vapor, or CO₂ during processing;
- The powder dispersal system must be improved so that a larger mass loading of powder can be dispersed in the aerosol without aggregation;
- A remaining fraction of error trajectories remain, in which particles traveling near the vane surfaces experience velocity dispersion and wall interactions that could introduce trace impurities of large particles into the separated fines;
- A more effective means of removing the fines from the separated aerosol flow is required (previous studies filter paper separation used µm filter paper to collect fines, not suitable for bulk flow).

The present work had as its goals to develop solutions to the first three of these issues. The powder was dispersed into a flow of N₂ gas obtained from boil-off of liquid nitrogen. The fluidized-bed dispersal system shown in Figure 4 was used to improve dispersal from 0.6 mg/liter (obtained with a rotating plate disperser in earlier studies) to 14 mg/liter, which is the limit at which particle-particle interactions should begin to interfere with separation. The threshold for particle size separation remained sharp and there was no evidence of aggregation during flow through the separator.

III. ERROR TRAJECTORIES IN BOUNDARY FLOW

A boundary sheath flow of powder-free N₂ was injected along the vane surfaces so that powder particles in the convergent laminar flow do not encounter the region of velocity shear near the vanes and also cannot impact the vane surfaces. The effect of the boundary sheath flow has been simulated using ANSYS CFX™; the results for particles with Stk = 2 are shown in Figure 5. Two effects are evident. First, heavy particles in the low-velocity flow near a vane surface can make a crossing trajectory, as exemplified in Figure 5a for Stk = 15. Second, particles very near the vane surface can scatter from the surface and be deflected into the major flow, as shown in Figure 5b for Stk = 2. Figure 5c shows an example of this latter phenomenon, observed by laser fluorescence on dye-tagged particles. The experimentally observed contamination fraction η of the major flow from these effects is shown in Figure 3 as a function of Stk. Such contamination would be unacceptable for the precursor powders for PIT superconductors.
Table 1. Superconducting precursor powder samples for separation studies.

<table>
<thead>
<tr>
<th>Powder</th>
<th>Client</th>
<th>Density (g/cm³)</th>
<th>Dₜ₀ (µm)</th>
<th>D₉₀ (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MgH₂</td>
<td>Ohio State U.</td>
<td>1.7</td>
<td>1.9</td>
<td>5.0</td>
</tr>
<tr>
<td>MgH₂</td>
<td>Ohio State U.</td>
<td>1.7</td>
<td>3.9</td>
<td>10.0</td>
</tr>
<tr>
<td>MgB₂</td>
<td>U. of Wisconsin</td>
<td>1.0</td>
<td>0.5</td>
<td>1.0</td>
</tr>
<tr>
<td>Mg</td>
<td>Hypertech</td>
<td>1.7</td>
<td>1.9</td>
<td>5.0</td>
</tr>
<tr>
<td>Sn</td>
<td>Supramagnetics</td>
<td>7.3</td>
<td>0.85</td>
<td>2.2</td>
</tr>
<tr>
<td>Cu₅Sn₄</td>
<td>Supramagnetics</td>
<td>8.9</td>
<td>0.85</td>
<td>2.2</td>
</tr>
</tbody>
</table>

IV. PARTICLE-FREE BOUNDARY SHEATH FLOW

Error trajectories can be suppressed by injecting a sheath flow of particle-free gas along each vane, indicated in Figure 5d. There are then no particles to experience the boundary effects and no defect trajectories.

The vane geometry is shown in Figure 6. Separate channels inject powder-free gas along the two boundaries of the inlet channel, corresponding to 5% of the inlet cross section. Sharp particle size separation was retained for mass loading up to 14 mg/liter, greater than any previous micron-scale aerosol separation process.

V. SEPARATION OF SUPERCONDUCTING PRECURSOR POWDERS

Samples of superconducting precursor powders were obtained from manufacturers and researchers, as detailed in Table 1. For processing superconducting precursors, dry nitrogen gas was used as the buffer gas, in order to avoid contamination from O₂, CO₂, or H₂O that would be present in air.

The size for the separation threshold for each powder was chosen by the client in each case: Dₜ₀ is the threshold particle diameter for 50% transmission into the minor flow; D₉₀ is the diameter for 90% transmission into the minor flow. The powders included:

- Sn and Cu₅Sn₄ used by Supramagnetics in the fabrication of Nb₃Sn wire;
- MgH₂ used in the development of a new route for MgB₂ synthesis at Ohio State University;
- Mg flake used in fabrication of MgB₂ at Hypertech Research;
- MgB₂ powder that has been sintered and high-energy-milled at the UW Applied Superconductivity Center.

The before/after particle size distributions are shown in Figure 7. Interestingly the powders that we obtained from superconducting material developers turned out to provide an excellent ‘acid test’ of the VI processing. One powder consisted almost entirely of ‘rocks’ much larger than the desired threshold; VI processing removed all of the rocks. Another sample consisted almost entirely of fines; VI processing passed 90% of the fines.

VI. SCALE-UP TO KG/HR THROUGHPUT

The VI separation technology is now being scaled up to accommodate kg/hr throughput. Two aspects of the VI design must be substantially modified.

The buffer gas must be recirculated through the separator flow path. A turbine will be used to recirculate the inert gas. A 2-dimensional array of vane assemblies has been designed that can provide ~100-500 g/hr throughput of the above PIT
powders within the 14 mg/liter mass loading that has been obtained successfully with the fluidized-bed dispersal system. The fines must be removed from the recirculating major flow gas using a more robust technique than the filter paper extraction that has been used in our prototype studies to date. Indeed removing µm-scale particles from an aerosol dispersion is a quite difficult proposition.

Electrostatic precipitation is normally initiated by producing ions in the gas flow. The ions are drifted transverse to the gas flow by applying an electric field to the side walls of the conduit. The ions can undergo charge-exchange interactions with particles in the gas stream, transferring net electric charge to the particles. The particles are then cleared from the gas flow as they pass through a further region of transverse electric field, which causes them to drift sideways into a collection region.

In previous technologies for electrostatic precipitation, the ions are produced by a corona discharge around an electrode that is sustained at high voltage within the conduit, or from the ionization of gas molecules by the beta or alpha decay particles of a radioactive source. In either case, the charging mechanism is effectively only for removing particles larger than a size of ~5-10 microns.

We plan to develop an electron-beam-assisted electrostatic separator, in which a beam of ~10 keV electrons is injected through a thin Si3N4 window and used to produce effective charging of particles even down to a fraction of a µm size.

VII. CONCLUSION

An inert-gas-buffered aerosol technology for zero-defect removal of particles over a selectable µm-scale threshold has been demonstrated. Its use to separate precursor powders for PIT superconductors has been performed. Provisions for upgrading the system to provide ~kg/hr throughput processing are being developed.

REFERENCES