PARTICLE SIZE DISTRIBUTIONS OF SEVERAL COMMONLY USED EJECTING AEROSOLS

F. L. Crosswy
Calspan Corporation/AEDC Division
Arnold Air Force Station, Tennessee
INTRODUCTION

The classical dilemma in wind tunnel laser velocimetry is the necessity to provide flow entrained particles large enough for detection by the laser velocimeter (LV) system yet small enough to closely follow the gas velocity gradients. The number density of proper size intrinsic particles is oftentimes inadequate for productive LV operations, especially in closed-circuit wind tunnels, so that particle seeding of the flow is required. An ideal aerosol generator for wind tunnel seeding purposes can be defined as one which has the following characteristics:

1. monodispersed particle size distribution
2. selectable particle size
3. controllable particle production rate
4. particles with large scattering cross section
5. particles with low mass density
6. non-toxic, non-contaminating and inert aerosol
7. long particle lifetime in the test environment
8. seed material readily available and reasonably priced

Several different processes can be exploited for seed particle generation purposes. These include:

1. fluidization of solid particles
2. atomization of liquids
3. vaporization/condensation of liquids
4. atomization of solid particle suspensions
5. atomization of solid material solutions
6. chemical reaction
7. combustion

In practice, all of these processes fall short of the ideal, with monodispersity being one of the more important but one of the more elusive requirements.

The particle size characteristics of several commonly used seeding aerosols were recently studied at AEDC. In general, undesirable polydispersed particle size characteristics were observed. However, reasonably narrow size distributions were produced by the atomization of polystyrene spheres and by the vaporization/condensation of dioctyl phthalate (DOP).

The purpose of this paper is to provide brief descriptions of the aerosol generation devices and aerosol particle sizing system used in this study and to provide a catalog of particle size distributions for the aerosols studied to date.
FLUIDIZED BED SEEDER

The apparatus used at AEDC to produce solid particle aerosols at low pressure (<100 psi) is shown in Figure 1. This device is referred to as a mechanically agitated, fluidized bed seeder. Solid particles in powder form are continuously stirred by a rotating hollow tube assembly. Carrier gas, usually air or nitrogen, flows through the tube assembly and jets into the powder through small holes to create the solid particle aerosol. Studies to date include aerosols derived from various powders of aluminum oxide, magnesium oxide and titanium dioxide.
LIQUID ATOMIZERS

Two types of liquid atomizers are often used to create liquid droplet aerosols for laser velocimetry. One of these, the Collison nebulizer (Ref. 1), is shown in Figure 2. A jet of air or other gas is used to shear a column of flowing liquid to create a large number of small liquid droplets. The larger droplets are usually eliminated by impact upon a solid surface just downstream of the air jet. The liquid column can be either pumped or aspirated into the air jet.

The second type of atomizer is the Laskin nozzle, a form of which is shown in Figure 3 (Ref. 2). As with the Collison nebulizer, a liquid droplet aerosol is produced by shearing an aspirated liquid column with an air jet.

Figure 2. Collison Nebulizer

Figure 3. Laskin Nozzle
Condensation of a vapor homogeneously dispersed in a carrier gas can yield a narrow size distribution of condensate particles. In addition, control of the mass fraction of vapor with respect to the carrier gas affords some control over the size of the condensate particles. These two principles are exploited in the vaporization/condensation seeder shown in Figure 4.

The atomizer in Figure 4 can be either the Laskin nozzle type or the Collison nebulizer type. The atomizer assembly includes a provision for introducing dilution carrier gas as a means for varying the vapor mass fraction. The polydisperse aerosol from the atomizer is passed through a 5-ft long, 2-in. diameter stainless steel tube. The top part of the tube is heated by an electrical heater tape capable of temperatures up to 900°F. The atomizer aerosol droplets are vaporized in this section of the tube. In the lower part of the stainless tube the vapor condenses to form an aerosol with a more uniform particle size distribution than that originally produced by the atomizer.

The AEDC vap/con seeder is based upon fundamental information provided in the scientific (Ref. 3) and commercial (Ref. 4) literature. However, the high operating pressure (1000 psi) and high particle production rate capabilities are based upon design condensations provided by Yanta (Ref. 2).
The system shown in Figure 5 was used to determine the particle size characteristics of the various seeding aerosols. This system consists of an ASAS-X particle sizer interfaced to a DEC 11/23 microcomputer system and Tektronix 4631 Hard Copy Unit. The ASAS-X is a laser light scattering device produced by Particle Measuring Systems, Inc. The particle diameter range from 0.09-3.0 microns is covered by four fifteen-bin instrument ranges of 0.09-0.195 microns, 0.15-0.3 microns, 0.24-0.84 microns and 0.6-3.0 microns. The ASAS-X is calibrated with precisely sized latex spheres so that particle size measurements, even for irregularly shaped particles or particles with a complex index of refraction, are in terms of equivalent latex spheres. The DEC 11/23 was programmed to display and tabulate the high resolution data from each of these four instrument ranges or to provide the wide-range, quick-look histogram data used in this paper.

The test aerosol was introduced into a large plastic bag (6 ft$^3$) which had previously been purged and filled with bone-dry nitrogen. Background particle counts using this scheme were typically less than 50, while aerosol particle counts were typically in the range from 50,000 to 800,000 counts. Although rarely necessary, the DEC 11/23 was programmed to subtract the background count from a test aerosol count.

Figure 5. Aerosol Particle Sizing System
METAL OXIDE POWDER AEROSOLS

Because of their high melting points, various metal oxide powders are often-times used for seeding high temperature flows. Table 1 compares the properties of several commonly used metal oxides. The MgO is attractive for its high melting temperature, the TiO₂ for its large index of refraction and the Al₂O₃ polishing powder because it is specified as being nominally sized.

Table 1. Physical Properties of Metal Oxide Powders

<table>
<thead>
<tr>
<th>Material</th>
<th>Index of Refraction</th>
<th>Density, gm/cc</th>
<th>Melting Temperature, °F</th>
</tr>
</thead>
<tbody>
<tr>
<td>MgO</td>
<td>1.74</td>
<td>3.58</td>
<td>5072</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>1.76</td>
<td>3.96</td>
<td>3660</td>
</tr>
<tr>
<td>TiO₂</td>
<td>2.6-2.9</td>
<td>3.7-4.1</td>
<td>3326</td>
</tr>
</tbody>
</table>

Alumina Powder Aerosols

The alumina (Al₂O₃) polishing powders are of interest for possible use in LV seeding applications since the manufacturer's specifications include a nominal particle size. Figure 6 shows the particle size characteristics of an aerosol derived from 1.0 micron alumina polishing powder. Note that no peak occurs at 1.0 micron while many agglomerates larger than 1.0 micron are evident as well as a large number of particles smaller than 1.0 micron. A polydisperse aerosol like this is undesirable for LV seeding purposes since the large agglomerates will not closely follow the flow velocity gradients while the very small particles, below the LV system detection limit, can only contribute spurious scattered light.

The particle size spectrum for 0.3 micron alumina polishing powder is shown in Figure 7. The agglomerate problem is not as bad as for the 1.0 micron alumina powder but the small particle problem is worse. Note, once again, that the size spectrum does not peak at the specified particle size.

The spectrum for 0.05 micron alumina polishing powder is shown in Figure 8. If individual particles are no larger than the 0.05 micron manufacturer's specification, then the entire spectrum within the range of the ASAS-X must be made up of agglomerates.

The "Super-Finish" alumina polishing powder is a relatively new product. The 0.3 micron version of this powder was recently evaluated for LV seeding purposes since the manufacturer's specifications indicate that this product exhibits a lessened tendency to form agglomerates compared to the standard polishing powder. However, a comparison of Figure 9 with Figure 7 shows that, for aerosol generation purposes at least, the Super-Finish powder actually has a greater tendency to form agglomerates. On the other hand, the Super-Finish powder exhibits a lower percentage of particles at the small particle end of the size spectrum and it also has a histogram peak in the vicinity of the specified 0.3 micron particle size.

The search for means to reduce the percentage of agglomerates in powder aerosols includes the use of additive flow agent materials. One flow agent that has been reported (Refs. 5-6) to be effective in reducing agglomerates in metal oxide powder aerosols is a hydrophilic material described as a flame phase silica. This
material is widely used to enhance the bulk flow properties of powdered foods, indus-
trial chemicals and pharmaceuticals (Ref. 7). A commercially available but pro-
prietary mixture of 1.0 micron alumina polishing powder and silica flow agent was 
obtained for evaluation. The aerosol particle size characteristic is shown in Fig-
ure 10. Comparison of this particle size characteristic with that of the 1.0 micron 
alumina without flow agent (Fig. 6) shows that this particular flow agent prepara-
tion is, at best, only marginally effective in reducing aerosol agglomerates. A mix-
ture of 0.7 micron alumina and silica powders was obtained from the same commercial 
source and evaluated. The size spectrum is shown in Figure 11. Again, the agglomer-
ate content is objectionable.

A mixture of 1.0 micron alumina polishing powder and 0.5 percent by weight of 
silica flow agent with a nominal particle size of 0.007 microns was prepared at 
AEDC and tested. The aerosol particles size characteristic was found to be virtually 
identical to that of Figure 10. A 5-percent mixture of the silica flow agent and 
0.3 micron alumina powder was also prepared and tested. This mixture ratio was 
entirely unsatisfactory since an increase in agglomerates was observed rather than 
a decrease.

Figure 6. Alumina Polishing Powder, Nominal Size - 1.0 Micron
Figure 7. Alumina Polishing Powder, Nominal Size - 0.3 Micron

Figure 8. Alumina Polishing Powder, Nominal Size - 0.05 Micron
Figure 9. Super-Finish Alumina Polishing Powder, Nominal Size - 0.3 Micron

Figure 10. Alumina Polishing Powder With Silica Flow Agent, Nominal Alumina Particle Size - 1.0 Micron
Fig. 11. Alumina Polishing Powder With Silica Flow Agent, Nominal Alumina Particle Size - 0.7 Micron
Magnesium Oxide Powder Aerosols

For this study, no magnesium oxide (MgO) powder could be found for which the manufacturer could quote a nominal particle size. Therefore, only the two readily available reagent grade MgO powders were evaluated. These two forms of MgO powder are referred to as "light powder" and "heavy powder". The aerosol particle size characteristics are shown in Figures 12 and 13. Note the high percentage of very small particles in the aerosols of both powders.

![Graph showing particle size distribution for MgO Reagent Grade Light Powder](image1)

**Fig. 12. MgO Reagent Grade Light Powder**

![Graph showing particle size distribution for MgO Reagent Grade Heavy Powder](image2)

**Fig. 13. MgO Reagent Grade Heavy Powder**
Titanium Dioxide Powder Aerosols

Several different titanium dioxide (TiO₂) powders were acquired for which the manufacturer furnished particle size specifications. These powders are produced for use as pigment additives for paint, paper, and plastics. The particle size range is quoted as 0.1-5.0 microns with most particles being in the range of 0.25 micron. The aerosol particle size spectrums for two of these powders are shown in Figures 14 and 15. The particle size spectrum for the plastic pigment powder (Fig. 14) was found to exhibit a peak in the size range 0.44-0.6 microns whereas the paint pigment powder produced an aerosol with a size spectrum peak in the same size range plus a large percentage of agglomerates and/or large particles. The specified particle size of 0.25 microns is not dominant in the aerosol particle size spectrum of either powder.

Fig. 14. TiO₂ Plastic Pigment Powder

Fig. 15. TiO₂ Paint Pigment Powder
Aerosol Flow Conditioning

The ever-present tendency of submicron particles to form agglomerates and the undesirable presence of very small particles have led to the use of various aerosol flow conditioning schemes in pursuit of the ideal monodispersed size distribution. Cyclone type separators have been reported (Refs. 6 and 8) as being useful for size fractionation of LV seeding aerosols. Marteney (Ref. 9) has described a dispensing nozzle with near-sonic conditions in the nozzle passage which was shown to be useful for breaking up agglomerates. The use of sonic flow in an array of 25 x 300 micron slits in the wall of a tube has been reported for breaking up agglomerates. A simple, circular sonic orifice was recently evaluated at AEDC for breaking up the metal oxide powder agglomerates. A TiO$_2$ powder size spectrum is shown in Figure 16 just before attachment of the sonic orifice assembly to the aerosol generator (Fig. 1). The particle size spectrum after deagglomeration by the sonic orifice is shown in Figure 17. These results are encouraging for such a simple procedure. However, deagglomeration is achieved at the expense of increasing the small particle number density. The conditions necessary for the onset of agglomerate breakup by aerosol interaction with a normal shock wave recently have been formulated by Forney and McGregor (Ref. 10).

TOTAL PARTICLE COUNT = 114652
SAMPLE TIME - 40 SECONDS

![Particle Size Distribution](image)

Fig. 16. TiO$_2$ Powder Aerosol Before Deagglomeration
Fig. 17. TiO$_2$ Powder Aerosol After Deagglomeration by a Sonic Orifice
The particle size characteristics of aerosols produced by burning cigarette tobacco and incense are shown in Figures 18 and 19. For the cigarette smoke, 94 percent of the particles were smaller than 0.32 microns. For the incense smoke, 97 percent of the particles were smaller than 0.32 microns.

Fig. 18. Cigarette Smoke

Fig. 19. Incense Smoke
AEROSOLS BY LIQUID ATOMIZATION

Liquid atomizers are simple to operate and produce stable particle number densities. Unfortunately, however, those tested to date at AEDC produce high percentages of very small particles.

The Laskin Nozzle

The particle size distribution for dioctyl phthalate (DOP) aerosol as generated by the Laskin nozzle is shown in Figure 20. The DOP droplets are seen to be polydispersed with a preponderance of small particles.

The Collison Nebulizer

The size spectrum for DOP in the Collison nebulizer is shown in Figure 21. The percentage of small particles is seen to be larger than the Laskin nozzle and interestingly, this spectrum is similar to that of the 0.3 micron alumina powder (Fig. 7). The size spectra for olive oil and soybean oil were also found to be similar to that of Figure 21.

Fig. 20. DOP in the Laskin Nozzle
Fig. 21. DOP in the Collison Nebulizer
ATOMIZATION OF SOLID PARTICLE SUSPENSIONS

A notable example of this technique for generating a seeding aerosol is the atomization of a water or methanol suspension of latex spheres. With a sufficiently dilute suspension, most of the atomizer droplets will evaporate to leave a single latex sphere. The analytical, reference grade, latex spheres are attractive because of their extremely narrow size distribution. However, this product is prohibitively expensive for LV seeding purposes. A reasonably priced product is the base material used in latex paint. The manufacturer's specified particle size range is 0.35 - 0.55 microns. A sample of this material was prepared by mixing 5 parts by volume of the latex sphere suspension with 95 parts water. The Collison nebulizer was then used to atomize this suspension. The resultant aerosol produced the particle size distribution shown in Figure 22. An identical distribution was obtained with a 5/95 mixture of latex suspension and methanol. The peak in the distribution is coincident with the manufacturer's specification. However, the overall distribution is disappointingly polydispersed.

Fig. 22. Latex Sphere Suspension in the Collison Nebulizer
The first laboratory tests of the AEDC vap/con seeder involved comparisons of spontaneous and nucleate condensation processes. Consistent with the findings of Liu and Lee (Ref. 3) the intentional introduction of condensation nuclei resulted in more repeatable particle size distributions. Figure 23 shows the size spectrum for a 10,000/1 solution of DOP and anthracene with no dilution flow. The anthracene, with a higher vaporization temperature than DOP, became condensation nuclei for the vaporized DOP. By adding a dilution flow of nitrogen, the size spectrum of Figure 24 was obtained which illustrates the shift of the size spectrum toward smaller particles. Finally, the 10,000/1 DOP/anthracene solution was diluted in the volume ratio of 1 part DOP/anthracene to 99 parts ethanol and used in the vap/con seeder without dilution flow. The resultant particle size spectrum shown in Figure 25 is seen to be close to the ideal monodispersed size distribution.

**Fig. 23.** DOP Without Dilution Flow in the Vaporization/Condensation Seeder
TOTAL PARTICLE COUNT = 283709
SAMPLE TIME - 39 SECONDS

Fig. DOP with Dilution Nitrogen Flow in the Vaporization/Condensation Seeder

TOTAL PARTICLE COUNT = 122731
SAMPLE TIME - 28 SECONDS

Fig. Ethanol and DOP in the Ratio 99/1 in the Vaporization/Condensation Seeder
CONCLUSIONS

During the course of the study reported here, no solid particle powder could be found which produced an aerosol with a narrow particle size distribution when fluidization was the only flow process used in producing the aerosol. The complication of adding particle size fractionation processes to the aerosol generation effort appears to be unavoidable. In this regard, a simple sonic orifice was found to be effective in reducing the percentage of agglomerates in the several metal oxide powders tested. A flame phase silica flow agent was also evaluated as an additive to reduce powder agglomerates. Marginally beneficial results were obtained for a 0.5/99.5 percent by weight mixture of the flow agent and metal oxide powder. However, agglomeration was observed to be enhanced when the flow agent percentage was increased to 5 percent.

Liquid atomization using the Collison nebulizer as well as a version of the Laskin nozzle resulted in polydispersed aerosols with particle size distributions heavily weighted by the small particle end of the size spectrum. An even more extreme weighting toward the small particles was noted for tobacco and incense smoke.

The particle size spectrum for reasonably priced latex spheres was more polydispersed than had been hoped, in view of the monodispersed distributions produced by the more costly analytic, reference grade latex spheres.

The aerosol particle size distributions produced by the vaporization/condensation seeder were closer to the ideal monodispersed aerosol than any of the other aerosols tested. In addition, this seeding approach affords a measure of control over particle size and particle production rate.
REFERENCES


