Some Issues on Powdered-Aerosol Handling in the Aerospace Shock Tube Facility

January 2001

Prepared by

M. J. TRAUM AND E. L. PETERSEN
Propulsion Science & Experimental Mechanics Department

Prepared for

SPACE AND MISSILE SYSTEMS CENTER
AIR FORCE MATERIEL COMMAND
2430 E. El Segundo Boulevard
Los Angeles Air Force Base, CA 90245

Contract No. F04701-00-C-0009

Engineering and Technology Group

DISTRIBUTION STATEMENT: Approved for public release; distribution is unlimited.

DESTRUCTION NOTICE: For classified documents, follow the procedures in DOD 5220.22-M, National Industrial Security Program Operating Manual (NISPOM), Paragraph 5, Section 7. For unclassified, limited documents, destroy by any method that will prevent disclosure of contents or reconstruction of the document.
SOME ISSUES ON POWDERED-AEROSOL HANDLING IN THE AEROSPACE SHOCK TUBE FACILITY

Prepared by

M. J. TRAUM and E. L. PETERSEN
Propulsion Science & Experimental Mechanics Department

January 2001

Prepared for

SPACE AND MISSILE SYSTEMS CENTER
AIR FORCE MATERIEL COMMAND
2430 E. El Segundo Boulevard
Los Angeles Air Force Base, CA 90245

Contract No. F04701-00-C-0009

DISTRIBUTION STATEMENT: Approved for public release; distribution is unlimited.

DESTRUCTION NOTICE: For classified documents, follow the procedures in DOD 5220.22-M, National Industrial Security Program Operating Manual (NISPOM), Paragraph 5, Section 7. For unclassified, limited documents, destroy by any method that will prevent disclosure of contents or reconstruction of the document.
SOME ISSUES ON POWDERED-AEROSOL HANDLING IN THE AEROSPACE SHOCK TUBE FACILITY

Prepared

M. J. Traum
Propulsion Science & Experimental Mechanics Department

E. L. Petersen
Propulsion Science & Experimental Mechanics Department

Approved

R. B. Cohen, Director
Propulsion Science & Experimental Mechanics Department
Laboratory Operations

The information in a Technical Operating Report is developed for a particular program and is not necessarily of broader technical applicability.
Abstract

A study of the characteristics and behavior of submicron glass powders is pertinent to Aerospace research on two-phase combustion chemistry in the shock tube, particularly as it relates to the combustion synthesis of materials. A brief literature review of previous work covering aerosol dispersion, particle handling, and previously attempted shock-tube particle-injection techniques was conducted. Utilizing gas-dynamic theory, a preliminary estimate of particle settling velocity was obtained. At a typical shock-tube fill condition of 0.01 atm at room temperature, the settling time for a 200-nm SiO$_2$ particle is approximately 170 seconds; long enough to perform a 2-ms shock-tube experiment but short enough for pre-shock particle settling in the interval between filling the tube and breaking the diaphragm to be a concern. In contrast, at atmospheric pressure, a 200-nm particle remains suspended in the shock tube for approximately 30 minutes. However, settling is probably not a factor for submicron particles within a 1-mm optical probe volume during the time frame of a shock-tube experiment. Finally, a review of the potential risks and hazards associated with particulate handling was also performed. Based on this review, it is recommended that the equipment, facility, and experimental procedures be updated to include the use of a fume hood/exhaust, protective clothing, a dry box for handling the particles, and an airborne-pollutant concentration measuring system to improve safety in the Shock Tube Facility when experimenting with submicron metal oxides.
## Contents

Abstract ................................................................................................................................. v

1. Introduction ......................................................................................................................... 1

2. Powder Physics .................................................................................................................... 2

3. Previous Work .................................................................................................................... 3
   3.1 General Applications ....................................................................................................... 3
   3.2 Shock Tube Applications ............................................................................................... 3

4. Settling Time ....................................................................................................................... 5
   4.1 Theory ............................................................................................................................ 5
   4.2 The Trends ..................................................................................................................... 6
   4.3 Implications .................................................................................................................... 9

5. Safety Concerns ................................................................................................................ 10
   5.1 Laboratory Hazards ...................................................................................................... 10
   5.2 Personnel Hazards ........................................................................................................ 10
       5.2.1 Inhalation .............................................................................................................. 10
   5.3 Particulate Matter ......................................................................................................... 11
       5.3.1 Acceptable PM-10 Concentrations .................................................................... 11
       5.3.2 Acceptable PM-2.5 Concentrations .................................................................... 11
   5.4 Measurement Methods ................................................................................................. 12
   5.5 Handling, Storage, and Preparation ............................................................................ 13
   5.6 Facility Improvements ................................................................................................. 13

6. Summary ............................................................................................................................ 15

References .............................................................................................................................. 16
Figures

Figure 1. The Mixing Chamber Shock Tube Particulate Aerosol Injection Technique .................. 4

Figure 2. Calculated terminal velocity at four different gas pressures. Gas is argon at a temperature of 300K ................................................................................................... 6

Figure 3. Settling time of SiO₂ particles as a function of particle diameter for two temperatures: 300 and 4000 K and two geometries: shock tube and optical beam. Gas is argon at 1-atm pressure ........................................................................................................... 7

Figure 4. Calculated settling time for particles in the 16.2-cm Aerospace shock tube over a range of 4 different pressures. Gas is argon at 300K ........................................................................................................... 8

Figure 5. Calculated settling time for particles within a 1-mm laser beam (or optical probe volume) over a range of 4 different pressures. Gas is argon at 300 K ........................................................................................................... 8

Tables

1. Properties of Powders Utilized in the Aerospace Shock Tube Facility ............................... 2

2. Exposure limitations for powder inhalation ........................................................................... 12
1. Introduction

The Aerospace Shock Tube Facility is currently used for taking measurements related to the combustion synthesis of glass materials such as SiO$_2$ and TiO$_2$. For certain experiments involving particle composition and spectroscopy, the material must be introduced in a powdered, submicron particulate form. The shock tube then heats the powder to a temperature range of 1000 to 4000 K behind the reflected shock wave. To achieve accurate measurements, the particles must be monodispersed uniformly inside the shock tube. Proper dispersion is complicated by particle settling under gravity and flow-induced drag effects. The settling that occurs between the tube’s uniform filling and the test could affect optical measurements, which are based on laser absorption, laser scattering, and particle emission within small imaging areas. In the method currently used at The Aerospace Corporation, the powder is sprinkled onto a plate, placed inside the shock tube and scattered by the dispersive force of the incident shock wave. Another dispersion method employed in the literature involves injecting a premixed gas/particle suspension into an evacuated shock tube uniformly before the tube is fired.

In addition to the problems associated with running experiments with solid- or liquid-phase aerosols, powder-handling precautions add important concerns to the experimental procedure. Safe use of fine particulates is of paramount importance to the health and safety of those using the Shock Tube Facility, particularly since powder-handling procedures are relatively new to the lab.

This report includes a brief review of previous work covering aerosol dispersion and handling, along with summaries of previously attempted shock-tube particle-injection techniques. Additionally, an estimate of particle-settling velocity, based on gas kinetic theory, is used to predict aerosol settling time in the shock tube as well as particle residence time in a laser beam or optical image volume. Finally, a summary of the potential risks and hazards associated with particulate handling is presented along with recommendations for comprehensive safety techniques and methods of hazard prevention.
2. Powder Physics

Current work on glass combustion synthesis focuses on SiO₂ and TiO₂ powders with individual particles typically of 200 nm in diameter or less. Preliminary studies discussed herein use SiO₂ as the baseline powder, but the general results should apply to both (and similar) substances. Particulate crystalline silicon dioxide, commonly called silica sand, alpha silica, or free silica, is a translucent granular powder that is odorless and insoluble in water. Titanium dioxide is typically seen in two forms: rutile and anatase. Table 1 compares the physical properties of the two substances. Both have similar melting and boiling temperatures, but the density of TiO₂ is nearly twice that of SiO₂.

Table 1. Properties of Powders Utilized in the Aerospace Shock Tube Facility

<table>
<thead>
<tr>
<th></th>
<th>Silicon Dioxide (SiO₂)</th>
<th>Titanium Dioxide (TiO₂)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting Temperature (K)</td>
<td>1710</td>
<td>1570</td>
</tr>
<tr>
<td>Boiling Temperature (K)</td>
<td>2677</td>
<td>2500 - 3000</td>
</tr>
<tr>
<td>Density (g/cm³)</td>
<td>2.65</td>
<td>4.23</td>
</tr>
<tr>
<td>Solubility</td>
<td>Hydrofluoric Acid</td>
<td>Concentrated Acid</td>
</tr>
</tbody>
</table>
3. Previous Work

The literature contains a number of papers focused on the general behavior of aerosol-laden flows as well as the introduction of powder substances into shock tubes.

3.1 General Applications

Kim et al. (1984) studied the sedimentation of aerosol particles in cylindrical tubes with various constrictions. In all cases, particle deposition occurred to some extent upstream of a constriction and to a far greater extent just downstream of a constriction. Larger particles were found to have a higher rate of deposition, and the change in pressure across an obstruction was found to be a significant factor in deposition rate as the paper noted that the higher the change in pressure across a constriction, the more particles were deposited. Tube size and the ratio of throat size to tube diameter had little bearing on the amount of material deposited and only affected the distance downstream of the obstruction where the maximum buildup occurred.

Ingebrethsen and Matijevic (1979) generated solid particulate aerosols by using sprays of a liquid solution containing the dissolved molecule of interest. The droplets were sprayed uniformly and then evaporated leaving uniformly dispersed solid spherical particles. Varying the rate of liquid evaporation via chamber temperature was found to control particle size.

Tu (1982) created fields of monodisperse, solid particulate aerosols of stearic acid, carnauba wax, ammonium sulfate, and ammonium bisulfate using a condensation aerosol generator. The system operated by vaporizing the desired compound and then forcefully diffusing it through a mixing chamber. The chamber contained a steady stream of low-temperature seeding particles. As the vapor and seeding nuclei mixed, particles condensed out of the vapor. It was found that varying the rate at which seed particles were introduced into the mixing chamber controlled particle size.

Mitchell (1984) generated ferric-oxide particles by dissolving the substance in water and then utilized a spinning top to disperse the solution into an evacuated chamber. Once created, the aerosol solution was evaporated leaving monodisperse solid, spherical particles. It was found that either changing the amount of solid dissolved in solution or varying the revolution rate of the device controlled aerosol concentration. However, the latter resulted in variations in the projected distance of the particles and was not preferred.

3.2 Shock Tube Applications

Lowenstein and Rosenberg (1978) injected pulverized coal dust into a shock tube in an optically thin and uniform manner using an entrainment method. Their mechanism was an impulsively retractable pneumatic piston with orifices drilled through the disk face. Coal dust was entrained in the high-velocity jets of the gas compressed behind the retracting piston and was dispersed into the test section.

A number of experimenters, including Roth et al. (1984) and Rajathurai et al. (1990), simply dispersed powder in a shock tube by placing it on a tray and allowing the incident shock wave to scatter the sample throughout the tube. Brandt et al. (1987) expanded on this idea by utilizing a secondary shock tube to disperse a powder while a primary tube shock-heated the mixture. Other experiments took advantage of pressurized premixing or settling chamber techniques, as demonstrated in Figure 1. Watson et al. (1967), Hooker et al. (1969), Hooker et al. (1970) and
Cann et al. (1982) used similar experimental systems where the aerosol suspension was blended, pressurized, and then blown into an evacuated shock tube.

Nettleton (1966) introduced potassium sulfate particles into a shock tube by allowing the incident shock wave to rupture a 60-micron-thick Melinex diaphragm coated with the particles. In 1974, Nettleton placed quartz-fiber mats into a shock tube and painted them with hydrocarbon droplets. The shock wave shattered the mats, vaporizing and dispersing the hydrocarbon droplets. Woodburn et al. (1974) developed a circulatory feeding system consisting of a blower and a solids loader. This system, adapted for a vertically mounted shock tube, took powder settled on the bottom and continuously fed it to the top, where it was sprinkled in and allowed to settle again. Further details on the use of the shock tube in powder and related experiments can be found in Petersen (2000).
4. Settling Time

4.1 Theory

In a typical experiment, the average particle diameter is about 200 nm or less. At these small sizes, Brownian motion tends to keep the powder airborne for extended periods of time if it is dispersed within a gas. Barring outside perturbations, the settling velocity of particles in a gas is well understood and characterized by a modified Stokes equation (Watson et al., 1967)

\[ V = \frac{1}{18} \frac{d^2}{\mu} \left( \rho_s - \rho_g \right) \left( 1 + 2A \frac{\lambda}{d} \right) g \]  

where \( V \) is the particle settling velocity, \( d \) is the particle diameter, \( \mu \) is the gas viscosity, \( \rho_s \) is the particle density, \( \rho_g \) is the gas density, \( g \) is the acceleration of gravity, and \( \lambda \) is the gas mean free path. The term \( 1 + 2A \lambda/d \) corrects for the effect of low pressures and/or small particle sizes when the mean free path of the gas molecules is on the same order as the particle diameter (Orr, 1966). The constant \( A \) is given by

\[ A = 1.26 + 0.45 \exp(-0.54d/\lambda) \]  

The gas viscosity, \( \mu \), can be determined using the Sutherland Approximation (White, 1991),

\[ \frac{\mu}{\mu_o} \approx \left( \frac{T_g}{T_o} \right)^{3/2} \frac{T_o + S}{T_g + S} \]  

where \( \mu_o \) is the effective viscosity constant, \( T_o \) is the effective temperature constant, \( S \) is the Sutherland Constant, and \( T_g \) is the gas temperature. For argon, the primary shock-tube gas, \( \mu_o = 2.125 \times 10^{-5} \text{ N-s/m}^2 \), \( T_o = 273 \text{ K} \), and \( S = 144 \text{ K} \). Finally, the gas mean free path, \( \lambda \), can be determined via the kinematic equation (Nace, 2000)

\[ \lambda = \frac{R_u T_g}{\sqrt{2 \pi d_g^2 N_a P_g}} \]  

where \( R_u \) is the universal gas constant, \( d_g \) is the atomic gas molecule diameter, \( N_a \) is Avogadro’s Number, \( T_g \) is the gas temperature, and \( P_g \) is the gas pressure. For argon, \( d_g = 2.9 \times 10^{-8} \text{ cm} \).

4.2 The Trends

As Equations 1-4 indicate that settling velocity is a function of gas temperature, gas pressure and particle diameter only, a significant representation of the expected settling behavior can be gleaned by examining various permutations of these variables. Figure 2 shows the calculated terminal velocity for a spherical SiO\(_2\) particle as a function of particle size and gas pressure. As expected, the settling velocity increases with increasing particle size, with a 200-nm particle
descending at a rate of $7\times 10^{-4}$ cm/s at 1 atm and a 2-μm particle falling at 0.07 cm/s. The effect of lower pressures is to speed up the terminal velocity; i.e., at lower pressures the gas molecules are further apart, offering less interference for the particles. For particles greater than about 100 μm, the effect of pressure becomes negligible.

Figure 2. Calculated terminal velocity at four different gas pressures. Gas is argon at a temperature of 300 K.

Of immediate interest is the settling time for particles within the shock tube and within the probe volume of a laser and/or optical setup. Figure 3 shows the calculated settling time over a range of particle diameters at 1 atm and two different argon temperatures: 300 K and 4000 K, reflecting the extremes in temperature between the initial fill conditions and a high-temperature experiment. For the shock tube curves, the settling time is defined as the time it takes a particle to fall from the center of the 16.2-cm shock tube to the tube wall (i.e., 8.1 cm). Similarly, the laser beam curves represent a particle falling from the center of a 1-mm optical probe out of the field of view (i.e., 0.5 mm). In general, the settling time decreases with increasing particle diameter, and Figure 3 indicates only a slight difference in settling time between the two temperature extremes.

The major trend of gas pressure versus settling time is indicated in Figures 4 and 5 for the shock tube and laser beam volumes, respectively. Following the idea that gas density is a main factor affecting settling time, the figures show that as the argon pressure (and hence density) increases, the particle settling time also rises over a range of particle sizes from 10 nm to 1000 μm.
Figure 3. Settling time of SiO$_2$ particles as a function of particle diameter for two temperatures: 300 and 4000 K and two geometries: shock tube and optical beam. Gas is argon at 1-atm pressure.

Figure 4. Calculated settling time for particles in the 16.2-cm Aerospace shock tube over a range of 4 different pressures. Gas is argon at 300 K.
4.3 Implications

As shock-tube measurements are taken under the assumption that the particles are uniformly distributed and not in motion, it is important to characterize the experimental time scales in which this assumption is valid. There are three specific phases of the experimental procedure where this consideration is important.

The first significant duration occurs between the filling of the tube and the initiation of the shock wave. For a typical uniform dispersion of 200-nm-diameter particles, at fill conditions of 300 K and 0.01 atm, it will take approximately 170 sec for half of the particles to settle to the bottom of the shock tube. Hence, for negligible particle settling, the time between filling and firing should be considerably shorter than 170 sec, perhaps on the order of a few seconds. As the current arrangement makes it impossible to fill and fire the shock tube on this time scale, two obvious solutions are available: 1) an automated system incorporating a double-diaphragm driver section could be designed to fill and fire the tube within the allotted time; 2) a continuous flow mechanism similar to Woodburn et al. (1974) in conjunction with a quick-firing scheme could be incorporated to eliminate particle settling altogether.

The second important time scale associated with particle settling is the length of the experiment itself, about 1-2 ms. A typical particle in argon gas at 4000 K and 1 atm will reside within a 1-mm laser beam approximately 40 sec, significantly more time than is necessary to take readings. Therefore, provided the particle dispersion is uniform when the diaphragm is broken, the effects of settling should not impact the data acquisition process.

The third significant time frame is after the experiment, where any unreacted particles, particle agglomerates, and/or particles formed during subsequent reactions can be suspended in the shock tube.
tube. This is a potential safety hazard if workers are exposed to the post-test gas containing suspended particulates. In this scenario, the conditions are 300 K and 1 atm, where a 200-nm particle can remain suspended for approximately 30 minutes or more, and a 2-µm particle remains floating for only 10 sec. Hence, one can assume that particles above about 1 micron settle soon after the experiment while smaller particles can remain suspended in the gas mixture. Further safety issues are covered in the following sections.
5. Safety Concerns

Several important considerations must be made when using silicon dioxide and similar powder under laboratory conditions. These include laboratory hazards, personnel hazards, measurement methods, and particulate-handling techniques. In most cases, SiO$_2$ is referenced, but powder-handling concerns are expected to be similar for powders other than silica. Unless specified otherwise, the material in this section was obtained from various Web pages referencing the standards and procedures of the Occupational Safety and Health Administration (OSHA), the Environmental Protection Agency (EPA), and the California Air Resources Board (ARB). Suggestions pertinent to the Aerospace Shock Tube Facility are also provided.

5.1 Laboratory Hazards

According to the manufacturer's safety sheet (SIRI, 2000), SiO$_2$ powder should never be exposed to hydrofluoric acid as silica will dissolve in this acid and produce the corrosive gas silicon tetrafluoride. In general, silica powder is not considered a flammable substance in the laboratory. However, silica ignition can occur when exposed to unusually strong ignition sources such as a blowtorch and in the presence of strong oxidizers such as F$_2$ and pure O$_2$.

If the material is spilled, the manufacturer's recommended technique for cleanup is a dustless vacuum or water method. Any approved solid-waste disposal method is acceptable to dispose of discarded powder.

5.2 Personnel Hazards

Silicon dioxide powder cannot enter the body through the skin, according to SIRI (2000). Regardless, impervious gloves should be worn when working with the powder to avoid local skin irritation. Additionally, skin should be flushed with copious amounts of water if contact with the powder is made to avoid accidental ingestion or eye exposure. If the substance is accidentally ingested, medical assistance should be sought immediately (although the exact hazards are not clear from the literature). To avoid eye exposure, ANSI-approved chemical work goggles must be worn when working with the powder. If the powder does contact the eyes they should be flushed with warm water for at least 15 minutes, and medical attention should be sought as needed.

5.2.1 Inhalation

Silicon dioxide most readily enters the body via inhalation. If the powder is accidentally inhaled, the victim should be removed to fresh air and given oxygen and artificial respiration as needed. Most of the chronic exposure symptoms and work-site standards set up by the government organizations are concerned with workers' exposure to silica-based (and other) powders at industrial manufacturing and construction sites. The resulting hazards should be less severe in the present shock-tube application but are included herein for completeness.

OSHA (1978) indicates that signs of long-term overexposure to silica include shortness of breath, difficulty breathing, coughing, phlegm production, and increased susceptibility to colds and respiratory infection. Individuals with medical conditions constituting damage to the lungs can experience aggravation to their injury from exposure. The chronic condition of pulmonary fibrosis (silicosis) can also result with long-term exposure. The progress of silicosis can be stopped, but cannot be cured or reversed. The incidence of tuberculosis is high among silicosis patients. Additionally, silicon dioxide powder is a known carcinogenic substance.
To avoid high concentrations of airborne silicon dioxide powder in a work environment, local ventilation should include a fume hood and collection fans or scrubbers at the source. Further details regarding the inhalation of powder are given in the following section.

5.3 Particulate Matter

Particulate matter consists of small liquid or solid particles floating suspended in the air. Particles less than 10 microns in diameter, called PM-10, are the most unhealthy as they are small enough to evade the body’s natural defenses and embed in the deepest part of the lung, the air sacs, according to the California ARB (1998). A defined subcategory of PM-10, which is made up of airborne particulate smaller than 2.5 microns, is called PM-2.5. Airborne silicon dioxide powder is of the form of PM-2.5.

Health problems associated with exposure to PM-10 and PM-2.5 begin as the body reacts to inhaled foreign particles. Particulate inhalation can increase the severity of asthma attacks, aggravate bronchitis and other lung diseases, and reduce the body’s ability to fight infections. While the total exposure to all particle sizes is sometimes important, the dominant safety hazard according to OSHA comes from particles that can be inhaled. Hence, the exposure limits presented below are for inhaled concentrations only.

5.3.1 Acceptable PM-10 Concentrations

The EPA and the California ARB have developed different concentration safety guidelines for PM-10. Both organizations define short-term and long-term exposure limits for PM-10. The federal 24-hour PM-10 criterion is exceeded when a day’s PM-10 concentration is greater than or equal to 0.150 mg/m³ of air. The federal annual standard is exceeded when the arithmetic mean of all 24-hour PM-10 concentrations is greater than or equal to 0.050 mg/m³ of air. The state’s 24-hour boundary is exceeded when a day’s PM-10 concentration is greater or equal to 0.050 mg/m³ of air. The state’s long-term standard is exceeded when the annual geometric mean for all 24-hour concentrations equals or exceeds 0.030 mg/m³ of air. The state standards apply to both indoor and outdoor environments.

5.3.2 Acceptable PM-2.5 Concentrations

The EPA also has guidelines on acceptable short-term and long-term exposure to PM-2.5. The federal 24-hour PM-2.5 exposure limit is exceeded if concentrations above 0.065 mg/m³ air are detected in a 24-hour period. The annual federal standard is exceeded when the arithmetic mean of all 24-hour PM-2.5 concentrations is greater than or equal to 0.015 mg/m³ of air.

A value known as the Permissible Exposure Limit, or PEL, is defined by OSHA for most common powders. For crystalline silica powder, OSHA mandates that exposure be limited to 0.10 mg/m³ of air per 8-hour workday. An alternate PEL for silica is defined as 2.5 mppcf (million particles per cubic foot) over an 8-hour workday. The PEL value is determined for a workplace environment by measuring the particle concentration over a known time interval and weighting on an 8-hour basis.

Table 2 summarizes the applicable exposure limits for inhaling small particulates. Included in Table 2, along with the silica, PM-10, PM-2.5 values are comparative values for coal dust and "particulates not otherwise regulated."
Table 2. Exposure limitations for powder inhalation

<table>
<thead>
<tr>
<th>Powder</th>
<th>8-hr Exposure Limit</th>
<th>24-hr Exposure Limit</th>
<th>Yearly Exposure Limit</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silica</td>
<td>0.1 mg/m³ or 2.5 mppcf</td>
<td>-</td>
<td>-</td>
<td>OSHA</td>
</tr>
<tr>
<td>Particulates not otherwise regulated</td>
<td>5 mg/m³ or 15 mppcf</td>
<td>-</td>
<td>-</td>
<td>OSHA</td>
</tr>
<tr>
<td>Coal Dust</td>
<td>2.4 mg/m³</td>
<td>-</td>
<td>-</td>
<td>OSHA</td>
</tr>
<tr>
<td>PM-10</td>
<td>-</td>
<td>0.150 mg/m³</td>
<td>0.050 mg/m³</td>
<td>EPA</td>
</tr>
<tr>
<td>PM-2.5</td>
<td>-</td>
<td>0.065 mg/m³</td>
<td>0.015 mg/m³</td>
<td>EPA</td>
</tr>
</tbody>
</table>

5.4 Measurement Methods

OSHA (1978) stipulates that employee exposure measurements should represent the actual breathing zone conditions for each employee. Any desired method of monitoring airborne silicon dioxide powder can be utilized provided the method is shown to be accurate to within plus or minus 25 percent for concentrations equal to or greater than the permissible exposure limit. In addition, the total sampling time may not be less than 7 hours.

The California ARB collects PM-10 samples over a 24-hour period on an 8-inch by 10-inch quartz fiber filter. Using a high volume sampler equipped with a size selective inlet, the sampling suction rate is between 36 to 44 cubic feet of air per minute. Each filter is weighed before and after sampling to determine the net weight gain of the sample. Collection of samples occurs from midnight to midnight every sixth day. Other methods including the dichotomous sampler, the tapered element oscillating microbalance, and beta attenuation monitoring are also used.

Particle monitoring can be performed on the Aerospace Shock Tube Facility using the recently acquired Scanning Mobility Particle Sizer from TSI, Model 3936 SMPS. One of the components of this system is a Model 3010 Condensation Particle Counter, which can be used to monitor the particle concentration within the laboratory air. The sampling probe should be placed in an area near the openings of the shock tube and/or particle-handling apparatus to determine the maximum particle concentration and resulting hazard level to workers. These measurements should be performed both with and without certain safety precautions to determine the relative effectiveness of the safety devices.

5.5 Handling, Storage, and Preparation

Satisfactory handling and storage procedures include use of an adequate dustless ventilation system. OSHA (1978) mandates that any storage procedure must ensure that exposure to airborne particulate does not exceed 0.10 mg/m³ of air over an 8-hour period.

Employees who may be potentially exposed to silica should be apprised at the beginning of their assignment of the hazards, relevant symptoms, appropriate emergency procedures, and proper conditions and precautions for safe use or exposure. It should be noted that preparatory stages of powder production such as crushing, grinding, pouring, transferring, or abrasive blasting could cause a dust overexposure hazard.
Keeping the powder contaminant-free is also important to maintaining experimental accuracy. Holden and Hooker (1965) noted that when their silica material was reduced to particles of submicron size, the increased surface area caused the powder to readily absorb gasses and vapors or particles of other contaminant material. They noted that when finely ground material was exposed to normal room atmosphere, it became contaminated with moisture and dust. Storing and manipulating particulate material only under dry-box conditions solved the problem.

5.6 Facility Improvements

Based on the safety standards reviewed above, it is recommended that a number of improvements be made to the Aerospace Shock Tube Facility and the associated experimental procedures to increase employee and laboratory safety. These procedures, along with all of the risks associated with handling powders, should be explained to all employees before they are allowed to participate in using the shock tube.

There are three situations wherein the worker may be exposed to potentially harmful levels of airborne particulates in the shock-tube laboratory: 1) when the shock tube is opened for cleaning and/or diaphragm changing; 2) when the powder sample is prepared and introduced into the system; and, 3) when a pressurized powder-delivery system is utilized (not currently employed), a failed gasket and/or user error could result in the expulsion of a particle cloud into the laboratory.

While the third scenario is unlikely, harmful results can be avoided by putting the pressurized vessels and valves within secondary containment. The first scenario presents the most likely exposure situation, since the shock tube must be opened at the endwall for cleaning after each experiment and at the diaphragm location prior to the next experiment.

To reduce the amount of particulate inhaled by employees, the common practice of pumping down the shock tube after each run to reduce airborne contamination should be strictly enforced. This procedure should remove a large fraction of the submicron particles since, according to Figures 2-4, these particles should remain suspended for up to 30 minutes or longer when the post-test pressure in the tube is near 1 atm. However, the repeated exposure of the pump(s) to nanoparticulates is frowned upon by the manufacturer; this is a matter of further investigation, including consultations with workers from laboratories that have been performing similar measurements in aerosol-laden environments for several years.) A ventilation hood and flexible ventilation nozzle should be mounted near the end of the driven section to purge remaining particles that may escape when the end-wall section is removed for cleaning, etc. Also a PM-10 and PM-2.5 monitoring system meeting the California ARB and Federal EPA and OSHA safety standards should be put in place to constantly screen the particulate concentration in the air (see above discussion on particle measurement).

While gloves are currently consistently worn when handling powders, their use along with protective eye goggles should be made mandatory whenever the shock tube is opened, loaded, emptied, or cleaned. In addition, an eyewash facility should be made available in the shock tube laboratory within a close proximity to the end of the driven section (an eyewash station exists in the general lab area already, but it is inconveniently located relative to where potential eye exposure might occur). Furthermore, telephone numbers for poison control as well as general emergency should be posted visibly near the telephone in the lab. An appropriate dustless powder-cleanup mechanism (a portable vacuum system is ideal) should be available within the lab for both accidental powder spillage and to clean up most of the powder residue in the shock tube.
To minimize the effects of moisture and contaminants, a dry box with a dustless ventilation system should be installed in the shock tube lab and a directive put in place stating that powders must be stored in the dry box when not in use. In addition, the dry box should be incorporated into the aerosol-injection scheme so that the powder can be introduced into the shock tube without being exposed to the air.
6. Summary

In support of Aerospace's current shock-tube research on the combustion synthesis of glass with SiO₂, TiO₂, and related powders, a review of previous work in particulate aerosol dispersion and summaries of previous shock-tube particle-injection techniques was performed. A theoretical estimate of particle settling velocity was made based on kinetic theory, which predicts aerosol settling time in the shock tube as well as particle residence time in a sub-millimeter laser beam or image volume. A synopsis of the potential safety hazards associated with handling powder was presented along with a number of suggestions for improvement.

The conclusions of this report can be summarized as follows:

Previous investigations have developed techniques for uniformly filling shock tubes with particulates using an external, pressurized particle-suspension volume.

1. The theoretical trends indicate that particle-settling time decreases with increasing particle size and increases with increasing gas density.

2. The settling time is too short for an experiment to occur without the advent of automated shock-tube injection and firing and/or a continuously circulating particle delivery system.

3. Settling time is probably not a problem for submicron particles within an optical probe volume during an experiment.

4. The airborne particle concentration should be measured in appropriate locations in the shock-tube laboratory.

5. Some improvements should be made to the current experimental procedure and equipment in order to satisfy safety requirements and powder quality. These include the addition of a movable fume hood/hose near the removable shock-tube endwall, a portable vacuum cleaner, a dry box for handling powders, and a clear procedure for handling powder and cleaning the shock tube.
References


Nave, C., “Mean Free Path,” [http://hyperphysics.phy-astr.gsu.edu/hbase/kinetic/menfre.html](http://hyperphysics.phy-astr.gsu.edu/hbase/kinetic/menfre.html), Department of Physics and Astronomy, Georgia State University, 2000.


