Condensed Zinc Particle Size Determined by a Time Discrete Sampling Apparatus

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This paper describes an experiment in which a novel sampling technique is employed to determine the size distribution of zinc particles originating through homogeneous condensation of zinc vapor expanding with a helium carrier gas through a supersonic nozzle. Decelerating a skimmed portion of the nozzle flow by mixing with quiescent air at matched pressure was found to be an effective method of sampling. The particles were collected on a cylinder shuttered in a way which permitted several time discrete samplings within a very short time span. Electron microscope analysis of the particles sampled showed a narrow distribution of sizes with 80% of the particles having diameters within a 100 Å range and a peak number of particles having diameters about 135 Å.

Nomenclature

D = particle diameter
P_c = stagnation chamber pressure
T_c = stagnation chamber temperature
$\bar{T}$ = jet centerline temperature nondimensionalized by temperature at entrance slit
$\bar{U}$ = jet centerline velocity nondimensionalized by velocity at entrance slit
$\bar{X}$ = distance from entrance slit nondimensionalized by entrance slit width
κ = mass fraction of zinc vapor in stagnation chamber
Φ = particle size distribution function (see text)

Introduction

WHEN a superheated vapor is expanded in a nozzle, the curve describing the expansion generally intersects the vapor pressure curve; at this point, the vapor becomes saturated (Wegener and Mack[1] give an excellent discussion of this phenomenon). If the expansion is rapid and the gaseous mixture is relatively "clean," sufficient surface will not exist for the condensation necessary to maintain equilibrium, and a supersaturated vapor will result. As the supersaturation continues to increase, small droplets will be formed from the vapor through a process of spontaneous (homogeneous) self-nucleation. As the nucleation rate increases, a point is usually reached where enough droplets exist to provide the surface necessary for the faster condensation through particle growth, and the supersaturated state "collapses" and returns the vapor to an equilibrium expansion.

Most of the previous research on condensation phenomena has been for either a one-component flow or a minute amount of vapor in a carrier gas and has dealt with condensation of nonmetals having surface tensions, latent heats, molecular weights, and vapor pressures which are widely different from those of the metals. In that these factors have considerable effect upon the condensation process, extrapolation of the limited existing data to a flow comprised of comparable quantities of condensable vapor and carrier gas is questionable, especially so when the condensable vapor is a metal.

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Index categories: Multiphase Flows; Subsonic and Supersonic Air-breathing Propulsion; Nuclear Propulsion.

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Because of newly developed applications, recent studies[2–6] have investigated the condensation of metal vapors. The use of metal vapors as the working fluid in turbines shows distinct advantages.[2] However, the condensation of these metal vapors would markedly affect such things as turbine efficiency and blade erosion. When metal additives are employed in chemical rocket engines, condensation rates of the metallic oxides are a factor in determining combustion rates, while condensed particle size and energy release exert a significant influence on the amount of thrust generated. Metal vapor condensation is used to produce the chargeable particles of a colloidal EHD Energy Convertor,[7] and the condensation of a metal or metallic compound has been proposed as a method of producing the extremely small and numerous particles which make up the working fluid in a colloidal-electrostatic rocket engine.[8] These applications all require a knowledge of the size of the particles formed in the condensation process (or the number of particles formed, which can of course be directly inferred from the particle size distribution for a given total amount of condensate). Almost all of the previous condensation work, both theoretical and experimental, has been concerned, however, with the onset of condensation and its effect on the flow parameters rather than with particle size.

Most of the experimental work in measuring particle sizes can be generally categorized under four basic techniques: 1) light scattering,[9, 10] 2) particle separation,[11–13] 3) particle sampling,[6, 8] and 4) mass spectrometry.[14, 15] The light scattering techniques generally assume a monodispersion and result in a mean particle diameter. Techniques yielding polydisperse size distributions by angular scattering distributions have not yet been proved. Particle separation techniques have been used mostly for large particle sizes (order of 1-μ diameter). Thomann,[11] however, inferred very small ice crystal sizes (approximately 14 Å diameter) from a Pitot tube separation technique. Some particles have been collected on a blunt body placed directly in a Mach 2 flow,[8] and particle size has been measured by direct observation of particles swept from a receiving tank.[6] The dearth of significant experimental data is understandable in light of the complexity of the problem.

The fact that most metals are solid at standard conditions makes sampling procedures particularly amenable to the study of metal or metal compound condensates. This paper describes an experiment conducted to determine, by means of a novel sampling technique, the size distribution of solid zinc particles originating through homogeneous condensation of zinc vapor expanding with a helium carrier gas through a supersonic nozzle.

Production of the Zinc Vapor-Helium Working Fluid

For the experiment described here, the metal vapor was generated in the University of Michigan's hotshot wind-tunnel facility.[16] The hotshot was easily capable of furnishing the energy

for the operating conditions necessary to obtain the required superheated zinc vapor-helium mixture. In addition, the hotshot provided a ready means of introducing the zinc vapor into the flow by means of the arcing process employed in the hotshot —a zinc fuse and zinc center electrode furnished the proper amount of zinc vapor (i.e., a mass fraction of approximately 0.75). The arcing process heated the mixture well into the zinc superheat regime to chamber pressures of near 4500 psia (zinc partial pressure of 700 psia) and chamber temperatures near 4500°K. Although the mixture cooled considerably during the tunnel blowdown time of 30 msec, the zinc was still in the superheated regime throughout the run time.

The gas-vapor mixture expanded through a conical nozzle with a total included angle of 15°. The throat insert is of beryllium copper with a throat diameter of approximately 0.100 in. Because of erosion, the throat diameter increases approximately 0.001 in. during each run and, in fact, increased from 0.102 to 0.121 in. over a period of 15 runs. The throat size was considered constant during any one run. The nozzle is protected from the arc by a baffle; a centerbody on the downstream side of the baffle was employed to reduce the volume of the region between the baffle and the throat. The nozzle exhausts into an evacuated test section which connects to a vacuum tank.

Condensed Particle Collection

All of the methods of particle size determination mentioned earlier were investigated; however, the most promising and straightforward means of determining particle size distribution seemed to be to simply catch the particles and measure them.

The most obvious requirement of any particle sampler must be that method of sampling affects the results either in a known manner or not at all. Thus, the high-speed flow of particles and gas entering a sampler must be decelerated, prior to deposition of the particles, in a way which decreases the particle kinetic energy while keeping the temperature low enough to prevent significant revaporization. The particle sampler (shown in Fig. 1) incorporated a very narrow slit (of order 0.010 in.) at its leading edge to produce a very narrow, two-dimensional freejet just inside the sampler.

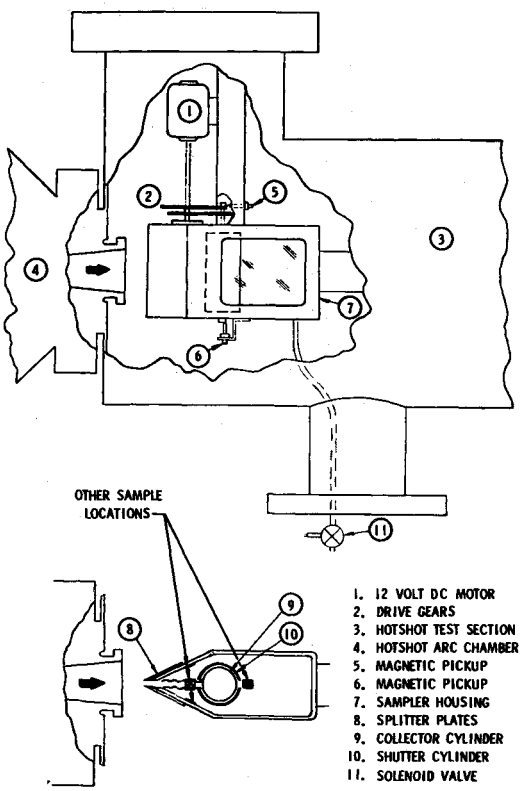

Fig. 1 Sampling apparatus mounted in hotshot tunnel test section.

The slit edge radius of curvature was approximately 2×10^{-5} in. and could be considered perfectly sharp. The half-angle presented to the flow was 22.5°. Thus, for a flow Mach number of over 2.3 (in helium), there was no possibility of the starting shock's not being swallowed when the sampler pressure was controlled. By matching the ambient pressure in the sampler to the static pressure of the jet, the jet was perfectly expanded, and the possibility of strong shocks was eliminated.

As the jet issued into the quiescent interior gas, a gradual deceleration by turbulent viscous mixing occurred (a Reynolds number of order 500 at the sampler inlet allowed the assumption of a turbulent jet).[17]

The sampler was located approximately $\frac{3}{4}$ in. downstream of the exit of the nozzle, and the particle collection was effected approximately 750 "slit widths" downstream of the entrance slit. By using a modification of a method derived by Abramovich[18] for determining the axial variation in velocity and temperature of a nonisothermal, two-dimensional compressible gas jet, it can be shown that the jet is effectively decelerated in this distance.[19] In that condensed particles are generally quite small (of order 150 Å diameter in this experiment), it can be assumed that they would decelerate with the stream.[20] Figure 2 shows, for a typical set of flow conditions, the variation in jet temperature and velocity as a function of nondimensional downstream distance ("slitwidths"). It can be shown that for zinc mass fractions up to 0.75, a normal shock located at the sampling cylinder would revaporize less than 5% of the condensed zinc in the flow.[19] It is apparent that the particles were effectively decelerated by the time they were collected.

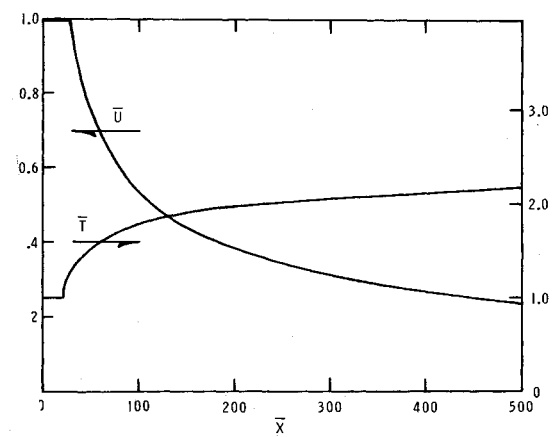

Fig. 2 Variation of flow properties in the two-dimensional jet inside the sampler.

To enable analysis with a transmitted beam electron microscope, it was necessary to limit the number of particles collected to no more than one "layer" and preferably much less. To accomplish this, a rotating collector with an attached mica sampling tape was used in conjunction with a concentric external shuttering cylinder as shown in Fig. 1. The shuttering cylinder possessed two opposite slots extending the full axial length of the cylinder and thus acted not unlike a camera's focal plane shutter.

A gear set, powered by a 12-v d.c. motor, drove the two cylinders—the outer shuttering cylinder at approximately 3000 rpm and the collection cylinder exactly 5 $\frac{1}{4}$ times faster. The factor of $5\frac{1}{4}$ spaced the zinc particle deposits at intervals of 225° on the collector cylinder and allowed (with the proper choice of shutter slot size) eight equally spaced deposits prior to deposit overlay. The eight samples were taken in four revolutions of the shutter or 80 msec.

After the sampling, a valve was opened which pressurized the interior of the sampler and effectively blocked the entrance of further particles or extraneous contamination. In service, the system was found to be very efficient when on one run the valve triggered prematurely, opening the sampler to atmospheric pressure prior to the start of the run. The resulting perfectly clean

sample tape proved the blockage efficiency of the interior pressure to the flow.

A plenum opened into the rear of the sampler preventing the entering gas sample from raising the pressure inside the housing during the first 40 msec. This was more than sufficient for the hotshot run time of 30 msec.

Electron Microscope Analysis

When the particles collected are too small to be seen under an optical microscope, an electron microscope must be employed. The instrument was a transmitted beam type where the beam must pass through the surface to be examined. At least part of the surface must then be transparent to the beam. In the case of the examination of small particles, the collection must then be sparse.

The procedure was based on the use of mica strips as a supporting surface. The mica was cleaved along lattice surfaces so that it was microscopically smooth and very thin. It was placed in an evaporator where a layer of carbon approximately 160 Å thick was deposited on it by evaporation and surface condensation at very low pressure. Such a carbon film was transparent to the electron beam. A much thicker layer would have been opaque, while a much thinner layer would have been structurally weak. The carbon coated mica was fastened to the collection cylinder in the sampler.

After the particles were collected, the mica strip was placed back in the evaporator where the surface was "shadowed" by evaporation of a thin film of chromium deposited at an angle to the surface. Chromium is opaque to the electron beam so that the shadows created permitted three-dimensional analysis of the particles photographed. The higher a particle stood above the collection surface, the longer was the shadow.

Copper grids provided the ultimate support for the film to be examined in the electron microscope. The film, consisting of carbon layer plus particles plus chromium layer, was removed from each piece of the mica, which had been cut to grid size, by floating it off the mica in water. As the film floated on the water, a copper grid was placed under it and the specimen was ready to be placed in the microscope and photographed.

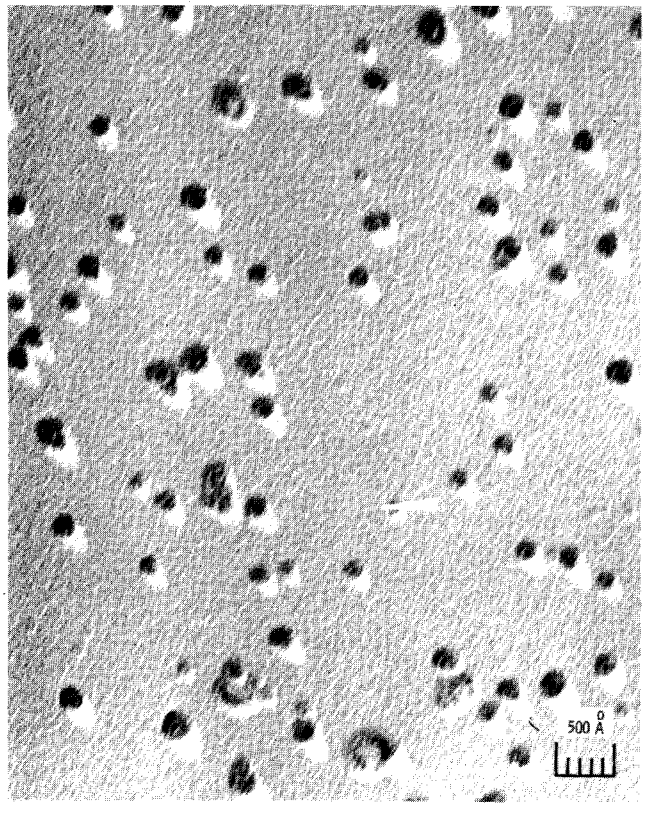

Fig. 3 Photomicrograph of collected particles (P_c = 2259 psia, T_c = 2338°K, κ = 0.785).

Photomicrograph Analysis

The electron photomicrographs were inspected qualitatively with respect to results from other runs and with an eye toward possible improvements in sampling technique. If the sampling was successful and good quality photomicrographs resulted, the particle size distribution was determined. To represent the size distribution graphically, a normalized probability function, Φ, was defined so that the integral of the function over a given particle diameter range will yield the fraction of particles having diameters within that range. The curve was determined by actual measurement of each particle on the photograph.

Results and Discussion

Figure 3 is an electron microscope photograph of a collection of particles obtained through methods outlined above. The condensed zinc particles appear round and dark. The background grain structure is the chromium layer used for shadowing. Each particle has a light oblong "tail" extending toward the lower right. This is the shadow cast by the particle in the chromium evaporated over the sample at an angle to the surface. The length of the shadow shows the height of the particle above the collection surface. It can be seen that the particles are indeed not only rounded but also close to spherical in shape. The size range is narrow (i.e., there seem to be no particles less than 60 Å and none greater than 250 Å). That this is not caused by the picture's small range of field can be seen in Fig. 4 which is a lower magnification photograph of the same sample. Here the lack of large particles becomes apparent.

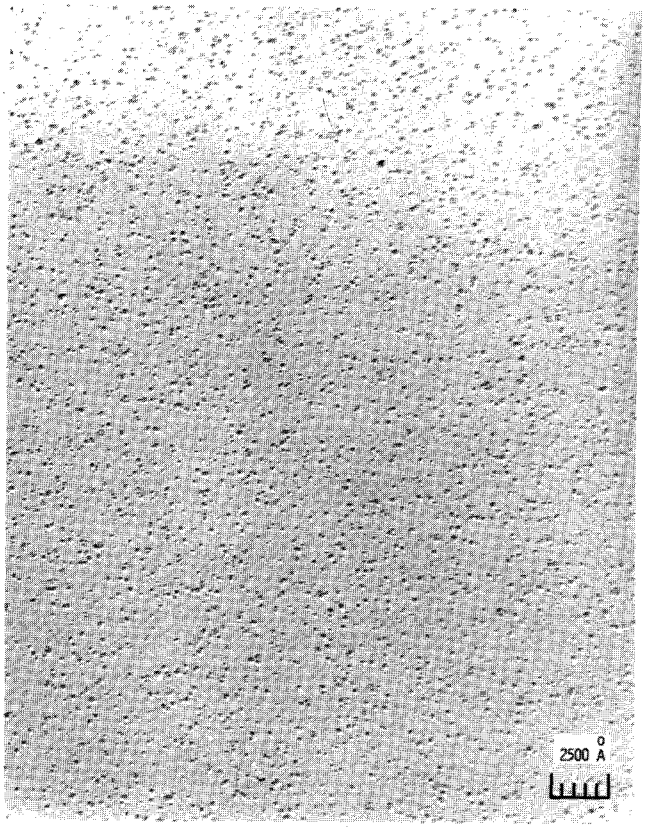

Fig. 4 Photomicrograph of collected Particles (P_c = 2259 psia, T_c = 2338°K, κ = 0.785).

The particle size distribution curve for the sample shown in Fig. 3 is given in Fig. 5. Φ, the distribution function defined above, is plotted against particle size. It can be seen to peak at a particle diameter of 135 Å, the most prevalent particle size. While plotting the data in this manner gives a very good qualitative description of the size distribution, the only quantitative data that can be easily obtained are the peak particle diameter and

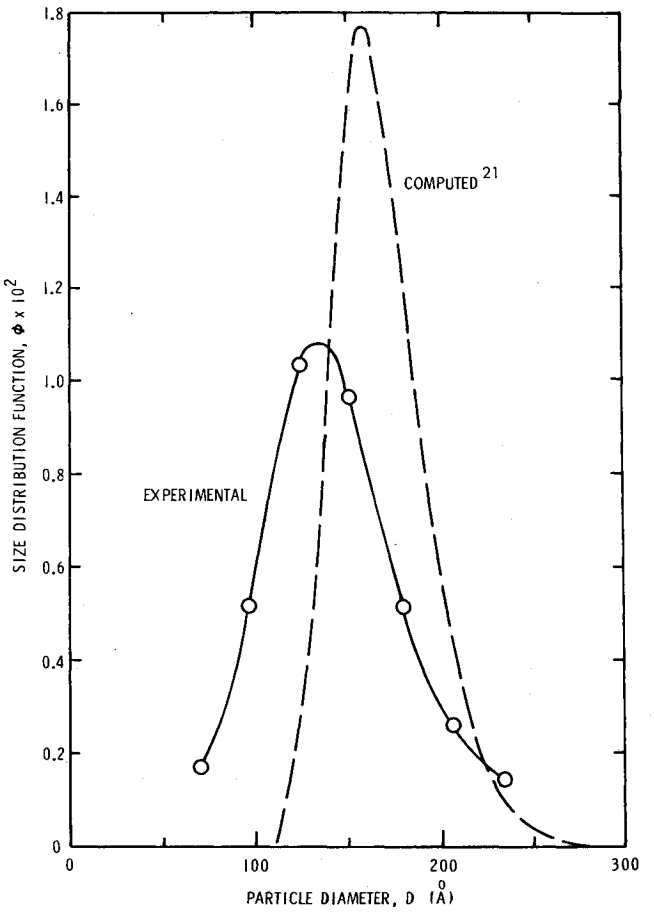

Fig. 5 Particle size distribution (P_c = 2259, T_c = 2338°K, κ = 0.785).

approximate width of the distribution. The results were also plotted in the manner of Figure 6 to give a more quantitatively descriptive evaluation. (This curve is simply the integral of the distribution curve.) It may be readily seen that the median diameter was 140 Å. As was indicated above, the curve is narrow: only 10% of the total number of particles have diameters greater than 195 Å and 10% have diameters less than 95 Å (i.e., 80% of the particles have diameters within a 100 Å range). This narrow distribution of condensate drop sizes about a median size agrees qualitatively (i.e., except for a particle size scale factor) with previous theoretical work and experiments.[9, 11] (These references deal with the condensation of water vapor in air with much smaller particle sizes.) A comparison was made between the experimental curve and one obtained from nonequilibrium calculations.[21] The agreement was found to be good—especially

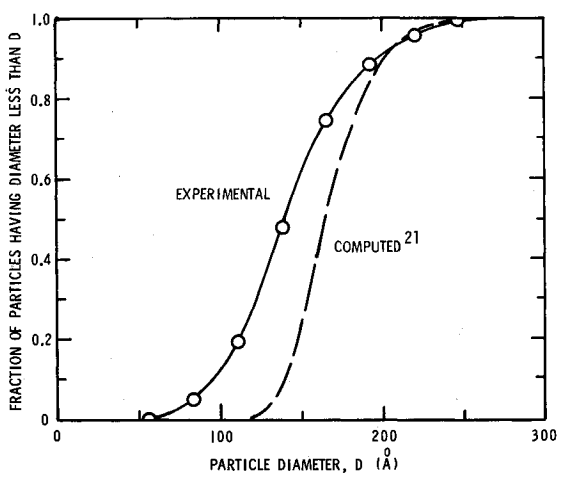

Fig. 6 Fraction of particles smaller than given diameter (P_c = 2259 psia, T_c = 2338°K, κ = 0.785).

the agreement between the peak diameters and the median diameters. As can be seen in Figs. 5 and 6, the calculated curve peaks at 155 Å; its median diameter is 165 Å, while the "80% limits" fall at 140 Å and 200 Å, yielding a 60 Å range of diameters. The difference in heights of the two distribution curves results only from the narrower distribution of the calculated sizes and the restriction that the integral of the curve equal unity. The excellent agreement lends added credence to the classical liquid drop nucleation model (which has recently been under increasing attack) used in the calculations of Ref. 21.

Figure 3 has been singled out for discussion because of its relatively high quality and optimum particle density (i.e., enough particles for a very good statistical sample but not enough to obscure one another). The size distribution of another sample taken during that same run, but much earlier (11.5 msec), is shown in Fig. 7. For comparison, the distribution of the sample taken at 22.4 msec (Fig. 3) is included. The 11.5 msec collection peaks at 125 Å (quite close to the same diameter as at 22.4 msec). However, the curve is characterized by a "bump" extending from a diameter of approximately 150 to 250 Å. The reason for the preponderance of particles in this size range at this sampling time is not clear. However, the facts that the chamber temperature was still high (3935°K) and that the arc was still established suggest the possibility of ionization effects.

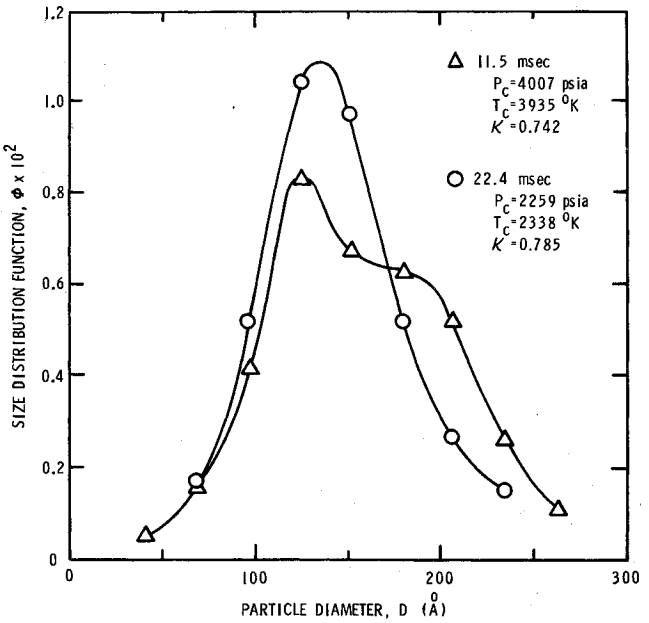

Fig. 7 Particle size distributions.

It has long been known that ionized particles will act as condensation nuclei in a supersaturated vapor. This is the underlying principle of the Wilson Cloud Chamber. Thus, because of the relatively low ionization potentials of metals and the very high temperatures reached in the arc chamber, the possible effect on condensation of ionization in the arc chamber is certainly a question which cannot be overlooked. Although no data for zinc are available, Solbes and Kerrebrock[4] have shown that nonequilibrium ionization effects on nucleation and condensation in potassium are minor at temperatures below 2000°K at 1 atm. A simple equilibrium ionization calculation on potassium vapor at these conditions yields an ionized mass fraction of 2.7×10^{-5}. Because zinc has a much higher ionization potential, stagnation conditions of 4650 psia and 4650°K and a zinc mass fraction of 0.75 are necessary to obtain a comparable ionization level. However, most of the measurements of particle size were made at stagnation pressures and temperatures approximately half that value. At the typical chamber conditions of 2500 psia, 2500°K, and a 0.75 zinc mass fraction, the ionized mass fraction of zinc is only 6.8×10^{-10}. Thus the equilibrium ionization level at stagnation conditions is five orders of magnitude less than an equilibrium level would be in the potassium

case. In that the higher density in the zinc flow would tend to minimize the nonequilibrium ionization downstream of the chamber, it seems reasonable that ionization, in toto, can be assumed to be negligible at that time during the run. Earlier in the run, when the temperatures are high and the arc is still established, it is not clear chat the ionization level would be. Heterogeneous condensation (i.e., condensation on foreign nuclei—such as ions) is usually characterized by earlier onset and larger particles and therefore could account for the bump in the curve. It should be noted that in the absence of this bump the curve is quite similar to the 22.4 msec curve. Recent theoretical work[22] has shown very similar results due to a coupling of heterogeneous and homogeneous condensation.

It is felt that contamination did not play a strong role in influencing any of the major findings of this study. The fact that the heavily eroded center electrode was made of zinc and that there was no evidence that the other end of the arc column established itself at any one point (other than the zinc fuse) would lend credence to the assumption that the only significant "contaminant" was zinc. It should be noted that hotshot contamination usually derives from only one electrode and the fuse, both of which were made of 99.99+% zinc. Further, considerable supersaturation of zinc vapor during the expansion[19] indicates negligible effects from contamination since contamination would have tended to cause a saturated expansion.

Figure 8 is the size distribution curve for a particle sample from another run but taken at similar stagnation conditions as the above. The experimental distribution curve compares favorably with the previous results.

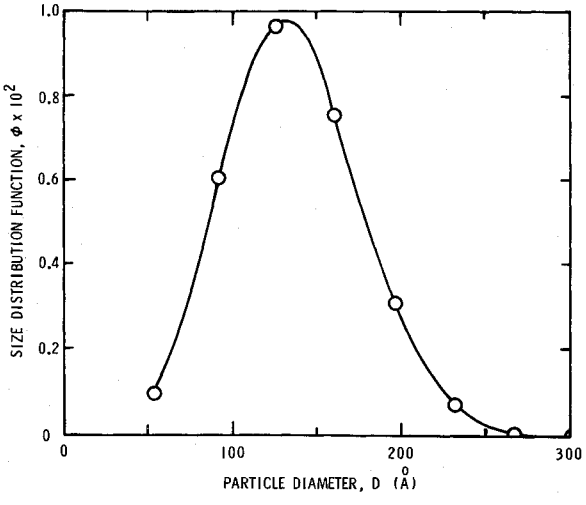

Fig. 8 Particle size distribution (P_c = 2453 psia, T_c = 2644°K, κ = 0.720).

Because of the exploratory and developmental nature of the study, the quantity of "good" data was limited, even though over 30 runs were actually made. Eighty percent of all particles collected in all runs were between 70 and 200 Å.

In a phase of the study not elaborated upon here but discussed in detail in Ref. 19, a sample of particles condensed in a Mach 5 nozzle was taken at a stationary location 6 ft downstream of the nozzle exit plane. Figure 9 shows that this sample includes considerable agglomeration. Few single particles were collected. This demonstates that many particle sampling methods that do not obtain the particles directly from an undisturbed freestream (e.g., such as "sweeping" a receiver tank following a run[6]) can be very susceptible to agglomeration effects.

In any collecting device, the question of collector efficiency is always important. In this case, because no analysis was intended concerning the total number of particles collected, the question became not "what is the efficiency?" but rather "how does the efficiency vary with particle size?" It was therefore necessary to obtain a reasonable estimate of whether the size distribution obtained from the rotating collector tape was indeed representative of the size distribution of particles entering the sampler. To accomplish this, samples were taken from elsewhere inside the sampler during each run.

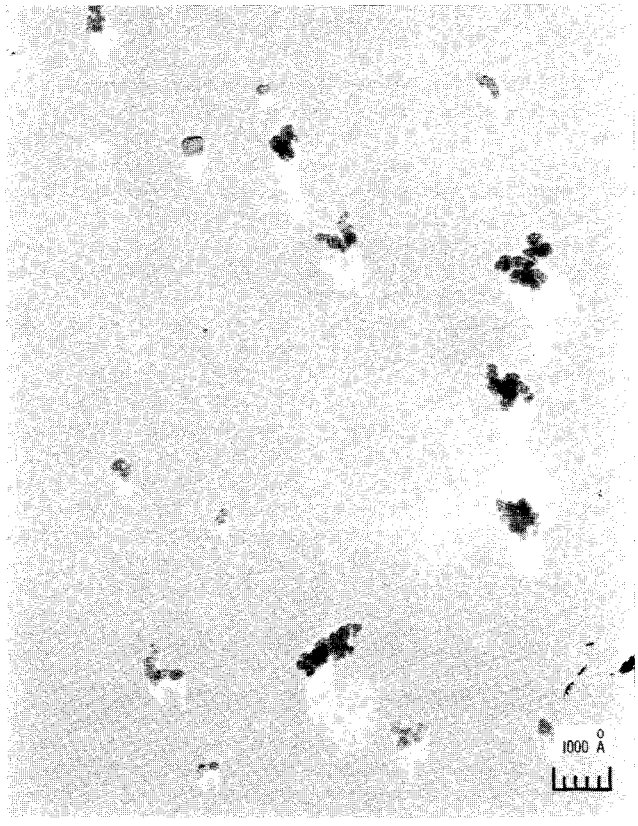

Fig. 9 Photomicrograph of particles obtained using the Mach 5 Nozzle; sample located far downstream of nozzle exit.

One location (see Fig. 1) was on the inside wall of the sampler, placed approximately on the centerline so that if particles were being "thrown" from the collector cylinder they would be caught at this point. The size distribution curve from this sample, shown in Fig. 10, is very similar to the collector's distribution curve of Fig. 5. (Both samples were taken during the same run.) Side wall samples taken during other runs yielded similar results. Although these samples were "time-integrated" (i.e., taken over the duration of the run rather than at a discrete time), it is still reasonable to assume that, since their distribution curves were similar to those obtained on the collector cylinder, collection was not weighted toward any particular size. The distribution curves obtained should therefore be representative of the free-stream particle size distribution.

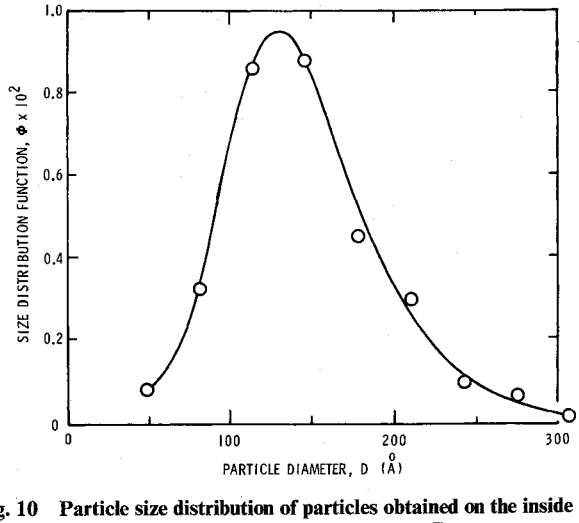

Fig. 10 Particle size distribution of particles obtained on the inside wall of the sampler (same run as Fig. 5).

The size distribution curves drawn from the experimental data are believed to be valid representations of the condensed flow to within a maximum possible error of ± 20 Å of particle diameter. This range of uncertainty is obtained from the particle size measurement reproducibility, which was a 0.02-in. range on a 132,000X photomicrograph.

One of the first questions to arise in a study of particle size concerns agglomeration. The region in which agglomeration could exert an effect on particle size is that distance extending from the saturation point, where particles first begin to be formed, to the particle solidification point, beyond which agglomerated particles would remain distinguishable and could be seen in the photomicrographs of the collected particles (i.e., see Fig. 9). In the present study, the total residence time of a particle in this region is typically only 13 μsec. In that the particles are small (of order 150 Å in diameter), it can be assumed there would be no velocity lag[20] and all the particles would accelerate with the gas. Brownian movement of the particles relative to each other could (if every particle collision resulted in coagulation) cause the number of particles to decrease by a factor of two during the 13 μsec.[23] This would result in a 25% increase in particle diameter. The assumption that every collision results in coagulation is very pessimistic; therefore agglomeration should tend to be considerably lower than this maximum amount. In this study, agglomeration effects were neglected.

Even though an expanding vapor becomes supersaturated, it is not evident, a priori, that homogeneous condensation will occur. The possibility exists that, if the vapor is expanded too rapidly, the nucleation process cannot produce enough nuclei to cause collapse of the supersaturated state or, if the vapor density is too low, the nucleation rate will never reach a sufficient level to cause collapse. Both conditions would result in a frozen expansion. Although preliminary calculations based upon classical nucleation theory indicated that condensation would occur in the nozzle, the theory in its present state of development was not considered reliable enough to furnish absolute proof that the condensed particles originated in the nozzle rather than the sampler. Therefore, to establish that condensation did, indeed, take place in the nozzle, Fastax photographs were taken perpendicular to a $\frac{1}{8}$-in. wide light beam traversing the flow between the exit of the nozzle and the sampler. Sufficient scattered light was seen from $t = 1$ msec throughout the run to establish the fact that condensation was occurring inside the nozzle during that time.

Summary of Results

An experimental investigation was conducted to determine the particle size distribution of solid zinc particles originating through homogeneous condensation of zinc vapor expanding with a helium carrier gas through a supersonic nozzle. Stagnation chamber pressures varied from 2250 to 4000 psia while chamber temperatures varied from 2350 to 4000°K. The zinc mass fraction was held at approximately 0.75.

The more important results of this study are presented below.

1) A particle sampler, which viscously decelerates the high-speed flow emanating from a supersonic nozzle, is an effective means of collecting very small particles while they are still held in suspension in the carrier gas.

2) The measured particle size distribution was found in general to be quite narrow with 80% of the particles having diameters within a 100 Å range. The distribution curve peaked at a diameter of 135 Å indicating the majority of particles clustered about that size.

3) The particle size distribution seemed to be relatively insensitive to the superheated stagnation conditions, but a great deal more data would be necessary to establish this fact.

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