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Production of Dry Powder Clots
Using Piezoelectric Drop Generator

Valeriy V. Yashchuk[a)]

Department of Physics, University of California, Berkeley, Berkeley, CA 94720-7300, USA

Alexander O. Sushkov[b)]

Department of Physics, University of California, Berkeley, Berkeley, CA 94720-7300, USA

Dmitry Budker[c)]

Department of Physics, University of California, Berkeley, Berkeley, CA 94720-7300, USA and Nuclear Science Division, Lawrence Berkeley National Laboratory, Berkeley, California 94720, USA

Eric R. Lee[d)]

Stanford Linear Accelerator Center, Stanford University, California 94309, USA

Irwin T. Lee[e)]

Stanford Linear Accelerator Center, Stanford University, California 94309, USA

Martin L. Perl[f)]

Stanford Linear Accelerator Center, Stanford University, California 94309, USA

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ABSTRACT

We have demonstrated that piezoelectrically driven, squeeze mode, tubular reservoir liquid drop generation, originally developed as a "drop-on-demand" method for ejection of microdrops of pure liquid or liquid suspensions of powdered bulk materials, can successfully operate with dry powder. Spherical silver powder with maximum particle diameter of 20 μm (-635 mesh) was loaded into and ejected from a 100 μm orifice glass dropper with flat piezoelectric disk driver. Time of flight experiments were performed to optimize the dropper operation parameters and to determine the size and velocity of the ejected particles. It was found that at certain values of the amplitude, duration, and repetition rate of the voltage pulses applied to the dropper piezoelectric disk, one can produce ejection of powder clots of a stable size, comparable with the dropper orifice diameter. In contrast to the dropper operation with a liquid, in the case of silver powder, a clot is not ejected at each high voltage pulse, but quasi-periodically with an interval corresponding to thousands of pulses. The application of the dry powder clot generation technique for injection of atoms into helium buffer gas at cryogenic temperatures is discussed.

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I. INTRODUCTION

At the core of many urgent fundamental and applied investigations is the ability to obtain long lifetimes of the ground state atomic polarization and correspondingly narrow atomic resonances (see, e.g. [1]). One of the most effective approaches to obtaining resonances with width as narrow as ~ 1 Hz [2, 3, 4] is to use alkali vapor cells with anti-relaxation (paraffin) coating ([5, 6, 7, 8]). An alternative method of linewidth reduction uses a buffer gas to decrease the rate of depolarizing collisions of atoms with cell walls ([9, 10, 11, 12]). Recently, with neon as the buffer gas, the so called coherent dark resonances with linewidths as narrow as 42 Hz in cesium [13] and 30 Hz in rubidium [14] were observed in vapor cells at room temperature.

In order to obtain ultra-narrow resonances with sub-Hz width, one can explore an extension of the buffer-gas method which relies on the properties of atomic scattering at low (cryogenic) temperatures, approaching the S-wave scattering regime, where collisional spin relaxation should be altogether suppressed. Theoretical estimates suggest that for the Cs-He case, a significant reduction of the relaxation cross-section, by a factor of $\sim 20-50$, may be expected already at liquid-Nitrogen temperatures [15, 16]. Recently, it has indeed been shown experimentally [17] that at temperatures below approximately 2 K, the spin relaxation cross-section of rubidium atoms in collisions with helium buffer gas atoms decreases by orders of magnitude in comparison with its room temperature value. It can be inferred from the data presented in Ref. [17] that relaxation times of minutes (corresponding to resonance widths on the order of mHz) or even longer can be obtained.

The crucial experimental challenge is creating, in the cold buffer gas, atomic vapor densities comparable to those in current room temperature experiments, i.e. of the order of $10^{10} - 10^{12}\ cm^{-3}$. The method of Refs. [17, 18] relies on photo-desorption of alkali atoms from the surface of a liquid He film inside the cell. It was possible to inject Rb atoms into the He gas by irradiating the cell with about 200 mW of Ti:sapphire laser radiation (750 nm) for 10 s. But it turns out that the injection efficiency decreases with repetition of the injection cycle. It was found that the efficiency recovered by heating the cell to room temperature and then cooling again. The authors of Ref. [18] also report that with their method they cannot inject Cs atoms which are of great importance among the alkali atoms for applications, in particular, for fundamental symmetry tests [19].

Currently we are investigating a significantly different method of injecting atoms into the buffer gas. It is based on laser evaporation of micron-sized droplets inside the cold He gas. A similar approach has been used for mass spectroscopy investigation of laser-induced volatilization and ion formation and for on-line chemical analysis of various micron-sized particles [20, 21, 22, 23]. A challenging problem for most of such experiments is constructing a droplet generator - a system for injecting micron-size solid particles into the apparatus. For example in [23], a complicated gas-dynamic system was used to introduce aerosols into the vacuum system of the time-of-flight mass spectrometer. However, such systems suffer from low injection efficiency, as low as 10^{-6}.

In the 1970ies, piezoelectrically driven, squeeze mode, tubular reservoir liquid drop generation was invented [24, 25] as a "drop-on-demand" method for ejection of liquid microdrops. Variations of the method were successfully used in searches for free fractional charge particles in pure liquids or liquid suspensions of powdered bulk materials [26, 27, 28, 29, 30, 31, 32, 33]. However, implementation of this method "as-it-is" is restricted to elements and substances whose melting temperature does not significantly exceed the operational temperature of the dropper's piezoelectric transducer [34]. In the present work, it is demonstrated that the method can successfully operate with a dry powder, generating a quasi-periodic chain of powder clots of stable size.

II. EXPERIMENTAL APPROACH

The design of the piezoelectric droplet generator used in our experiment has been described in detail elsewhere [31, 32]. The dropper consists of a pipette-shaped glass tube open at the top and narrowed down to an ejection orifice of 100 μm diameter at the bottom, see Figure 1 and inset therein. A piezoelectric transducer ring (outer diameter 1 in; inner diameter 1/4 in; thickness 0.1 in) made from lead zirconate titanate is attached to the lower portion of the tube with epoxy adhesive. The dropper is loaded with approximately 0.1 cc of spherical silver powder of maximum size 20 μm (-635 mesh) [35].

The experimental arrangement used to investigate and optimize the dropper operation parameters while it is loaded with silver powder is shown schematically in Figure 1. Electrical pulses from the high-voltage pulse generator cause the piezoelectric transducer ring to contract radially on the glass, forcing a powder clot to be ejected vertically downwards from the dropper orifice. The most consistent powder clot ejection was achieved for the high-voltage pulse width of $10-11$ μs and pulse repetition frequency of $450-470$ Hz. The pulse amplitude, V_p, could be varied in a wide range (300 to 500 V) without significantly affecting the powder clot ejection.

We found that the dropper operation with dry powder significantly differs from the original drop-on-demand regime where a liquid drop is generated at each high voltage pulse. In our experiment, a clot is generated quasi-periodically with many pulses necessary to produce one clot, and with a strong ejection dependence on the pulse repetition frequency. At optimum pulse repetition frequency of ≈ 460 Hz, powder clots are formed with some periodicity described by the Gaussian distribution of delay times between consequent drops with the mean time of 4.4 s and the standard deviation of 1.0 s.

Once the powder clot is ejected from the dropper orifice, it falls under the influence of gravity and air resistance. On its path, the clot intersects two parallel laser light beams, positioned one under the other, under the dropper orifice, and the scattered light signal is used for the time-of-flight measurement. The two light beams, each of horizontal width $\simeq 10$ mm and vertical thickness $\simeq 1$ mm, are formed by splitting the light beam from a diode laser (wave length 780 nm; output light power 20 mW). The vertical separation of the beams is $\triangle L = 21$ mm, kept constant throughout the experiment. Scattered light is detected with a photodiode and a high impedance preamplifier. The photodiode is equipped with a 780 nm interference filter reducing the noise from background room light. The detector overall integration time is $\simeq 0.2$ ms. The scattered light signal as a function of time has two adjacent peaks, corresponding to detection of light scattered while the powder clot passes through each of the laser beams. This signal is recorded with a digital oscilloscope (Tektronix TDS 410A), a typical trace is shown in Figure 2. For each such trace, by subtracting the positions of the two peaks, we calculate $\triangle t$, the time of flight of the powder clot between the two laser beams. By varying the falling height L (the distance between the dropper ejection orifice and the first laser beam) one can measure the $\triangle t(L)$ dependence. For large enough L this dependence flattens out, as the clot velocity approaches its limiting value, v_t, given by the Stokes Law:

$$v_t = \frac{2g\rho_{clot}a^2}{9\mu}, \tag{1}$$

where a is clot radius, ρ_{clot} is clot density and $\mu = 1.7 \times 10^{-5} Ns/m^2$ [36] is dynamic viscosity of air at normal conditions. From the distribution of the terminal velocities v_t the distribution of the clot "structure parameter" $\alpha = \rho_{clot}a^2$ can be deduced.

III. EXPERIMENTAL OBSERVATIONS

For each value of the falling height L and for each ejection pulse amplitude V_p, scattered light traces (as in Figure 2) for approximately 20 powder clots are recorded and the distributions of the obtained times of flight are plotted. Figure

3 shows histograms representing such distributions for two particular values of L and for $V_p = 420$ V. For $L = 121$ mm the observed values of $\triangle t$ are smaller than for $L = 19$ mm, corresponding to acceleration of the clots as they fall downwards. The spreads of the flight times are relatively small, indicating that powder clots of rather stable size are ejected from the dropper.

Each of the distributions is fit with a Gaussian

$$G(\triangle t) = \frac{const}{\sqrt{2\pi\sigma_\tau^2}} \exp\left(-\frac{(\triangle t - \tau)^2}{2\sigma_\tau^2}\right). \tag{2}$$

The mean time of flight τ and the spread σ_τ for each value of L and V_p are extracted from the fits.

The dependence of τ on L, measured at $V_p = 350,\ 420$, and 490 V is shown in Figure 4. No regular dependence of τ on V_p is observed for any L. To produce a fit to the data we solve the equation describing the downward motion $y(t)$ of the clot under the influence of gravity, g, and the air resistance, which we assume to be proportional to velocity, in accordance with the Stokes law [37]:

$$m\ddot{y} = mg - 6\pi\mu a\dot{y}. \tag{3}$$

The relationship between the clot's velocity, v, and the distance, L, it fell is

$$L = \frac{v_t}{g}\left[v_t \ln\left(\frac{v_i - v_t}{v - v_t}\right) + v_i - v\right], \tag{4}$$

where v_i is initial velocity and v_t is terminal velocity for $t \to \infty$, see Eq. (1). From Eqn. (3) one also derives the expression for the clot velocity v at the position of the top laser beam in terms of the experimentally determined time of flight τ:

$$v = v_t - g\frac{\tau - \triangle L/v_t}{1 - e^{-g\tau/v_t}}. \tag{5}$$

Substituting Eq. (5) into Eq. (4) gives the formula for fitting the relationship between L and τ.

The fitting results are shown on Figure 4 with a solid line, corresponding to the following values of the fitting parameters:

$$v_i = (24 \pm 12) \quad cm/s, \quad v_t = (92 \pm 2) \quad cm/s. \tag{6}$$

The initial velocity is significantly smaller than the terminal velocity of the clots. However, even for the smallest possible falling height L, the acceleration in the gravitational field is too large to extract the small initial velocity of the clots with small relative uncertainty.

Having deduced the terminal velocity v_t of the falling powder clots, one can calculate the mean value of the clot structure parameter $\alpha = \rho_{clot} a^2 = 7.2 \cdot 10^{-6}$ kg/m from Eq. (1). The spread of α can be estimated from the time of flight distribution (shown in Figure 3) for large value of L: $\sigma_\alpha \approx 0.3 \cdot 10^{-6}$ kg/m. The narrow distribution of the structure parameter can be thought of as evidence of the stable size of the ejected clots and strong binding of powder particles composing the clot.

To deduce the distribution of the clot radii a, one needs to make assumptions about the powder packing volume fraction within the clots. The observed stable size of the clots allows one to suppose that the volume fraction for the clots is the same as for the powder inside the dropper. As it was pointed out in [38], a random assembly of spherical uniformly-sized particles can be packed with a volume fraction, η, anywhere from $\eta \simeq 0.55$ to $\eta \simeq 0.64$, depending on how the container is filled. The reiterative vibration pulses applied to the container tend to compact the particles to the steady state value of the volume fraction $\eta \simeq 0.64$ (see, e.g. [39, 40]). Visual observations of the ejected clots were made by collecting them on a glass plate, positioned below the dropper orifice, and examining them under a microscope. The clots consist of about thirty spherical powder particles of about 20 μm diameter and much smaller particles whose combined volume is approximately one tenth of the clot volume. Therefore it is natural to assume packing slightly more compact than the steady state value, accounting the contribution of the smaller particles. We use the value $\eta = 0.7$ giving the clot density $\rho_{clot} \sim 0.7\rho_{Ag}$, where $\rho_{Ag} = 10.5 \times 10^3 kg/m^3$ is the density of bulk silver. This gives:

$$a \approx 31\mu m, \qquad \sigma_a \approx 1\mu m, \tag{7}$$

where the spread has been estimated from the structural parameter spread σ_α.

The estimated mean diameter of the ejected powder clots $2a \approx 62$ μm is comparable to the dropper orifice diameter of 100 μm. The ejected clots have very stable size and consist of powder particles bound strongly enough to keep the clot from disintegrating in flight.

IV. DISCUSSION

We have demonstrated that the piezoelectric liquid drop generation technique, originally used for searching for free fractional charge particles in liquids [30, 32], can successfully operate with a dry powder. In time-of-flight experiments with spherical silver powder of maximum particle diameter 20 μm (-635 mesh), it was found that at certain values of the amplitude, duration, and repetition frequency of high voltage pulses applied to the dropper piezoelectric disk, one can produce powder clots of stable size, $\sim 62\ \mu$m, comparable with the dropper orifice diameter. In contrast to the dropper operation with a liquid, in the case of the silver powder, a clot is not ejected at each high voltage pulse, but quasi-periodically with the Gaussian distribution of the time intervals between the consequent drops with the mean time of 4.4 s and the standard deviation of 1.0 s. The interval corresponds to thousands of high voltage pulses (consistent with the "inverse logarithmically slow" density relaxation in a vibrated granular material discussed for instance in [39, 40]) and the clot generation process resembles water dripping from a slightly open tap. The most puzzling question that remains is the origin of forces which bind the powder particles in the clot. One possibility is sticking by the possible residual moisture condensed on the powder particle surface. In order to verify the binding mechanism, we are planning the similar experiment in dry and inert atmosphere.

The obtained stable parameters of the powder clots, simplicity of their generation, a large range of both atomic and molecular substances available in the powder form, and the absence of a suspension liquid determine the broad spectrum of possible applications of the described method, see for example [41, 42]. The method is particularly suited to injection of atoms into the buffer gas via laser evaporation. Dry powder clot generation eliminates possible substrate-sample interferences in the experiment and decreases laser power transfer to the buffer gas. Estimates show that a 500 mJ, 10 ns-long, 2 mm beam diameter initial pulse from a YAG laser (1-micron wavelength) will cause the clot to explode into a collection of small clusters [43]. A subsequent light pulse will evaporate these clusters, creating dense atomic vapor in the buffer gas. Large residual drops will quickly leave the interaction region (compared to the expected polarization relaxation time of minutes).

As mentioned in the Introduction, with He buffer gas at cryogenic temperatures, it may be possible to obtain atomic resonances with widths on the order of mHz or even narrower. If such narrow resonances are realized, this could open exciting possibilities for a new generation of fundamental symmetry tests including, for example, a "traditional" experiment, searching for parity- and time reversal invariance-violating permanent electric dipole moment (EDM) of a free atom (see, e.g. [19]). Silver atoms have an EDM enhancement factor (the ratio of the EDM of an atom to that of a free electron) of ≈ 51 [44], which is only a factor of two smaller than ≈ 114 for cesium [45]. For gold atoms, the enhancement factor is even larger: ≈ 250 [46, 47]. Some molecular materials also available in powder form could be systems of interest for fundamental symmetry tests (see, e.g. [48]). Another possibility is to search for the electron EDM by measuring magnetization of a para- or ferro-magnetic bulk material induced by a strong applied electric field at low, ~ 50 mK, temperatures [49]. (An experiment of this type was first proposed by F. L. Shapiro in 1968 [50] and carried out by B. V. Vasiliev and E. V. Kolycheva in the end of the 1970's [51].) In this experiment, narrow resonances may be used to realize an extremely sensitive atomic magnetometer that would be used to detect the induced magnetization.

In conclusion, we note that in references [52, 53], it was demonstrated that the fluid ejector based on a flextensional transducer that excites axisymmetrical resonant modes in a clamped circular membrane (see, e.g. [54, 55]), can work as a solid particle ejector with talcum powder of $9 - 18$ μm size. This could be an alternative technique for the metal powder clot generation which deserves further investigation.

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[a] E-mail: yashchuk@socrates.Berkeley.edu

[b] E-mail: alex000@socrates.Berkeley.edu

[c] E-mail: budker@socrates.Berkeley.edu

[d] E-mail: erlee@slac.stanford.edu

[e] E-mail: itlee@slac.stanford.edu

[f] E-mail: martin@slac.stanford.edu

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CAPTIONS

Figure 1: Schematic diagram of the time-of-flight experimental arrangement used to determine the size, initial and terminal velocities of the ejected powder clots. IF - interference filter; PD - photodiode; PA - preamplifier. Inset shows an enlarged photograph of the dropper tip.

Figure 2: A typical time-of-flight spectrum measured with the following parameters of the high voltage pulses applied to the dropper piezoceramic transducer: amplitude $V_p = 420$ V; pulse duration 10.5 μs; repetition frequency 460 Hz. The distance between the dropper tip and the center of the first laser beam is $L = 19$ mm, the laser beam separation is $\Delta L = 21$ mm. The width of the peaks corresponds to the laser beam width of approximately 1 mm. The structures seen on the wings of the first scattered light peak are due to the light intensity distribution in the laser beam. The height of the peaks is determined by the location of the clot in the laser beam, the clot size and structure, and the gain of the preamplifier. From the peak signal-to-noise ratio (which corresponds to the clot with ~ 60 μm diameter; see Section III), one can estimate the lower size limit for clot detection of ~ 10 μm, which is smaller than the characteristic size of the powder particles.

Figure 3: The histograms represent the distributions of the flight times measured at $L = 19$ mm and $L = 121$ mm ($V_p = 420$ V) (see the data shown on

Figure 4). The dashed lines are the Gaussian fits with: (a) expectation value $\tau = 35.6$ ms, variance $\sigma_\tau^2 = (0.5)^2$ ms^2, and (b) $\tau = 25.9$ ms, $\sigma_\tau^2 = (1.1)^2$ ms^2 (see Eq. (2)). The observed narrow spreads of the flight times suggest that the generated powder clots have a rather stable size.

Figure 4: Dependence of the mean time of flight, τ, on the distance between the dropper tip and the center of the first laser beam, L. The solid line represents the results of fitting based on the procedure described in the text. The fitting gives the terminal clot velocity $v_t = (92 \pm 2)$ cm/s and the initial velocity $v_i = (24 \pm 12)$ cm/s. For each distance L, the measurements at the different high voltage pulse amplitudes, $V_p = 350,\ 420,$ and 490 V were performed. No regular dependence of the τ on the V_p was observed.

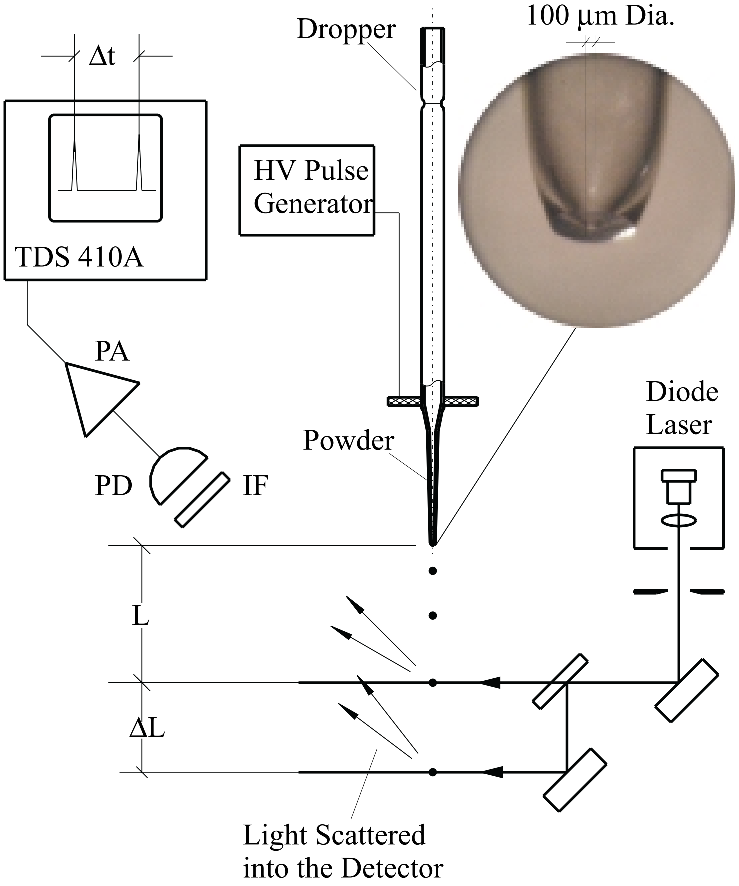

Figure 1: V. V. Yashchuk, et al "Production of Dry Powder Clots..."

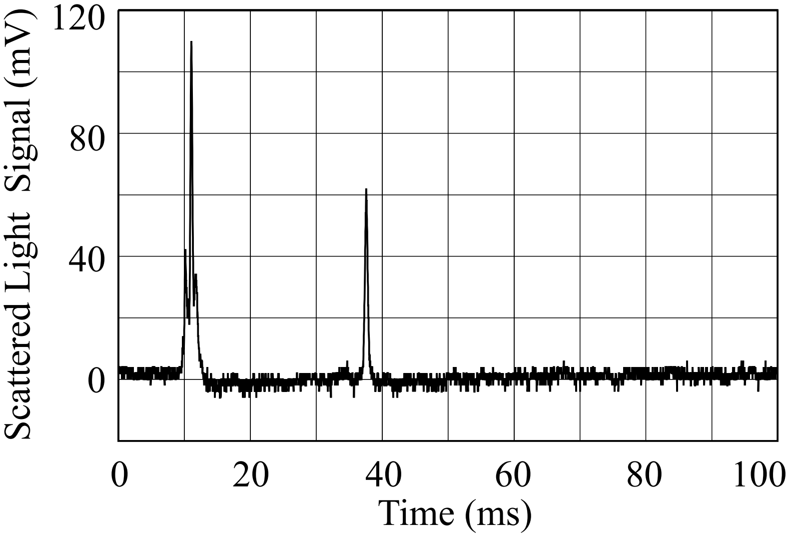

Figure 2: V. V. Yashchuk, et. al., “Production of Dry Powder Clots...”

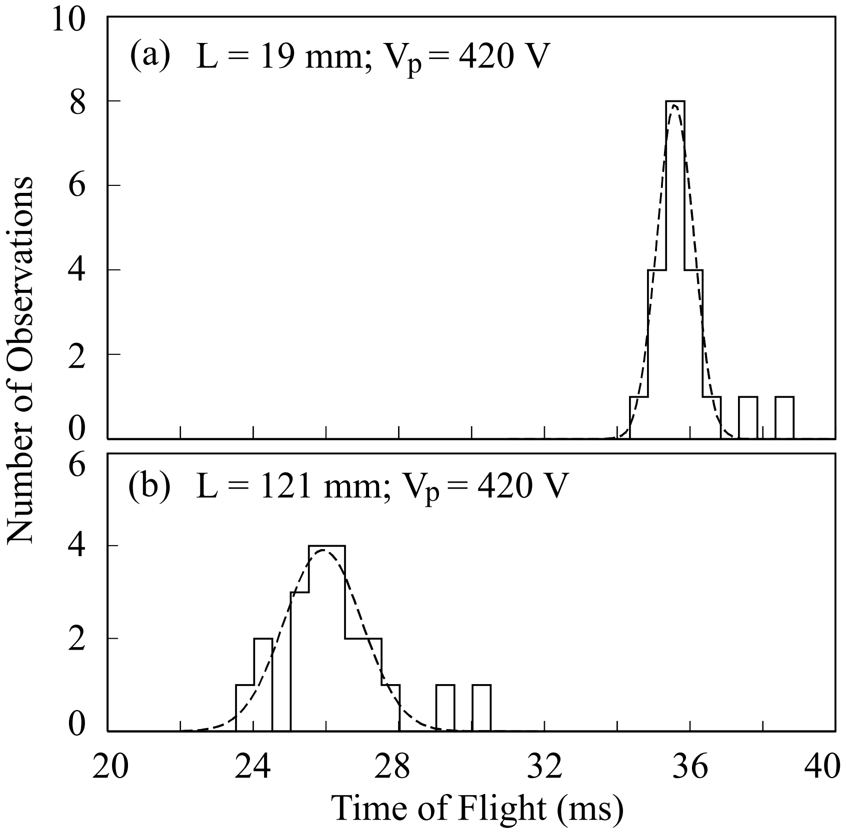

Figure 3: V. V. Yashchuk, et. al., “Production of Dry Powder Clots...”

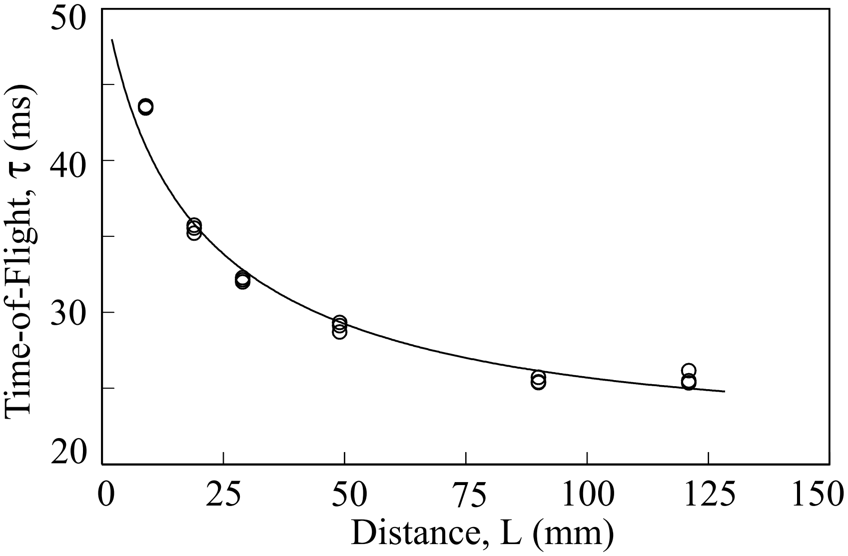

Figure 4: V. V. Yashchuk, et. al., "Production of Dry Powder Clots..."