7-1-1994

Simple Apparatus for Producing Single Liquid Drops

Dhiren Panda

D. G. C. Robertson
Missouri University of Science and Technology, davidrob@mst.edu

Recommended Citation

D. Panda and D. G. Robertson, "Simple Apparatus for Producing Single Liquid Drops," *Review of Scientific Instruments*, vol. 65, no. 7, pp. 2388-2394, American Institute of Physics (AIP), Jul 1994.
The definitive version is available at https://doi.org/10.1063/1.1144692

Simple apparatus for producing single liquid drops

Dhiren Panda and David Robertson
Generic Mineral Technology Center for Pyrometallurgy, 215 Fulton Hall, University of Missouri–Rolla, Rolla, Missouri 65401

(Received 20 October 1993; accepted for publication 31 March 1994)

A simple experimental apparatus has been used to produce individual mercury and sodium amalgam drops in the size range of 0.3–2.5 mm diam. A high resistance to the flow of liquid was provided by a capillary tube. The cross section consisted of a 0.20 mm o.d. tungsten wire placed in a 0.25 mm capillary bore. By this method, flow rates of 1 μl/s and lower were obtained, which enabled the formation of drops as small as 1 mm diam in a time interval of 20 s. The drops were formed at a plastic tip, and on reaching the desired size, were stripped off by a stream of argon. An optical sensor was used to detect the growing drop and to switch on the argon stream at the desired time. The growth of the drops at the tip was observed using an image analysis system which showed good agreement between the observed and calculated drop sizes at various instants. The consistency in size from drop to drop, for any particular setting of the apparatus, was evaluated by analyzing magnified pictures of the drops as well as by measuring their weights. The variation in the drop size was found to be within $\pm 2\%$ of the mean value.

I. INTRODUCTION

Several methods have been used by previous researchers to produce small drops.[1–5] The simplest method consists of forming drops at a fine tip by allowing the weight of the drops to overcome the surface tension forces.[1] The smallest drops that can be formed by this method are of the order of 1 mm diam. One way of producing drops smaller than 1 mm diam is by blowing off drops being formed at the tip by a constant flowrate of gas.[2] This method has been used to produce drops as small as 0.3 mm diam with consistent size. In another method, a stream of liquid issuing out of a fine capillary is subjected to transverse/longitudinal oscillations resulting in a stream of drops.[3–5]

The above devices produce a stream of drops. Elaborate arrangements are then required to separate individual drops from the main stream.[3–5] In the present apparatus, a high resistance to the flow of mercury/amalgam was created by placing a wire in a fine capillary bore, resulting in flowrates lower than 1 μl/s. The drops were formed at a plastic tip, and were stripped off by a gas jet on reaching the desired size. By this method, drops in the range of 0.6–2.5 mm diam could be produced individually whenever required, instead of a stream of drops. The apparatus was used to produce a wide size range of drops without any elaborate change in the setup (i.e., without requiring the replacement of tips, etc.). This flexibility coupled with a high degree of reliability makes the apparatus suitable for use in laboratories.

II. THEORY

The flow rate required to produce drops in the size range of 1 mm diam in a time interval of around 20 s, has to be of the order of 0.003 μl/s. A capillary with a very small cross section was used as a high resistance to get extremely low flow rates in the present apparatus. The volumetric flow rate through an annulus is given by[6]

$$Q=\frac{\pi \Delta P D^4}{128 \mu L} f(k), \tag{1}$$

where Q is the volumetric flow rate, ΔP is the net pressure difference, D is the diameter of capillary bore, μ is the viscosity of the fluid, and L is the length of the annulus. The function $f(k)$ is given as follows:

$$f(k)=\left((1-k^4)-\frac{(1-k^2)^2}{\ln(1/k)}\right), \tag{2}$$

where k is the ratio d/D and d is the o.d. of the wire.

In Eq. (1) we have defined ΔP as the net pressure difference. There are two different components of the net pressure difference that were considered separately in order to determine the effective flow rate. The first component is the applied pressure, which was maintained at a constant value by regulating the pressure over the reservoir of metal (described later). The second component, termed as the capillary

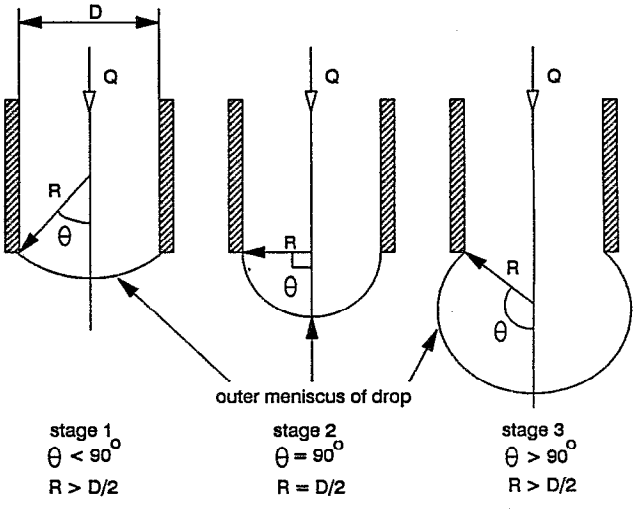

FIG. 1. The three stages in the growth of a drop at a tip.

 0034-6748/94/65(7)/2388/7/$6.00

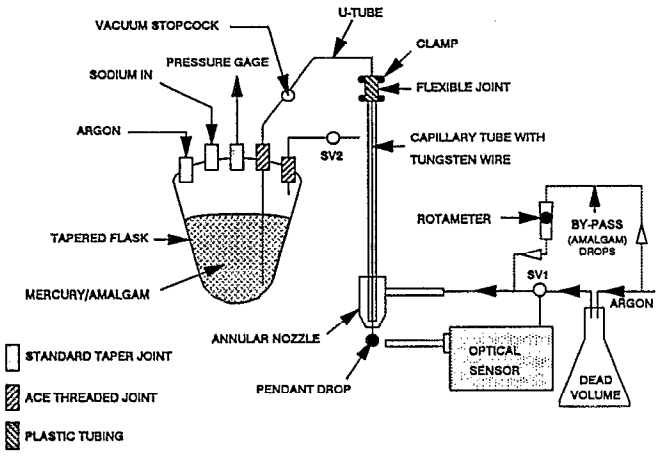

FIG. 2. Schematic diagram of the single drop apparatus.

pressure, is due to the surface tension of the drop that formed at the tip. Since the capillary pressure is inversely proportional to the diameter of the drop, this component of the net pressure difference became significant in comparison to the applied pressure, for smaller sized drops. As the size of the drop changed during its growth, this component of the net pressure difference was constantly changing. More important though was the fact that this pressure acted in opposition to the applied pressure and so helped in reducing the flow rate. The relationship between the components of the net pressure difference can be described as

$$\Delta P = P_{\text{appl}} - \frac{4\sigma}{D_d(t)}, \tag{3}$$

where P_{appl} is the applied pressure, σ is the surface tension of the liquid (mercury), and $D_d(t)$ is the instantaneous diameter of the drop.

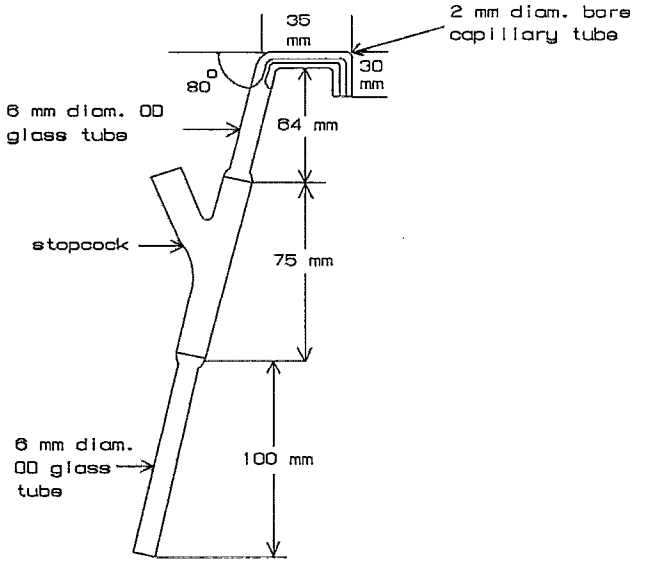

FIG. 3. Sketch of the U tube used in the apparatus.

For calculation purposes, it was assumed that the drop grew from a near-planar surface. The drop was considered to have a spherical cap profile in the initial stages of its growth. The angle subtended by the spherical cap with the vertical axis (θ in Fig. 1) was less than 90°, and the radius of the drop at this stage was larger than the radius of the tip. As the drop grew larger, θ increased while the radius of the drop decreased, until the drop radius equaled that of the tip, at which stage θ was a right angle. The radius of the drop was a minimum at this point, so the capillary pressure ($4\sigma/D_0$) was a maximum, which implied that the net pressure difference (ΔP) reached a minimum. Further growth in the drop resulted in an increase of θ and radius simultaneously. The overall effect was that the net pressure difference went through a minimum and then slowly built up as the drop grew in size. Since flow rate is directly dependent on the net pressure difference, the flow rate also underwent a minimum.

A program was written to calculate the various parameters such as the net pressure difference, flow rate, and instantaneous diameter of the growing drop. From this it was observed that the first stage lasted for a very short period of time while the third stage was predominant during the growth of the drop. The three stages in the growth of the drop and the related pressure–time relationship are shown schematically in Fig. 1.

The major problem in the use of very fine bores and annular spaces to increase flow resistance is the possibility of entrapment of pockets of air (gas). It has been observed by Liggieri *et al.*[7] that the entrapped pockets of air expand and contract with the change in the net pressure and thereby induce instabilities during the growth of a drop. These instabilities result in a sudden growth in the drop size with the forcible detachment of the drop from the tip. This phenomenon was also observed in the initial experimental trials with the annular bore. In later work the annular space was evacuated before filling it with the liquid to make sure that there were no entrapped air pockets.

III. DESCRIPTION OF THE APPARATUS

The apparatus consisted of a tapered flask with five ports (ACE Glass Inc., NJ), which was used as the reservoir for

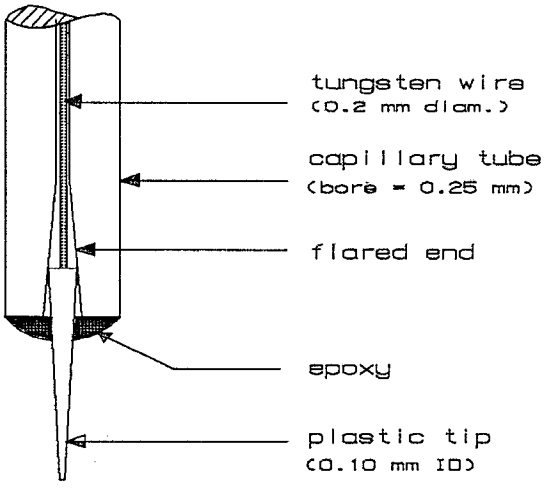

FIG. 4. Sketch of the tip used in the apparatus.

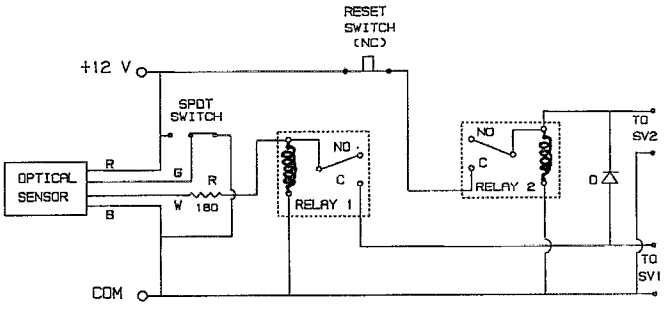

FIG. 5. Schematic diagram of the electrical circuit.

mercury (Fig. 2). Three of the five ports were of the standard taper joint type (14/20), which served as the ports for gas supply, addition of mercury, and an outlet for the pressure gauge. The remaining two ports were of the standard ACE thread type (No. 7) which allowed for the use of glass tubes, at the same time providing a good seal with the use of "O" rings. One of them was used for the "U tube" (Fig. 3) through which the metal flowed under pressure to form drops, while the other port was used as an outlet for the gases through a solenoid valve (SV2). The flask was held rigidly in position by a suitable clamping arrangement and was supported on a wooden platform.

The U tube consisted of a vacuum stopcock with a Teflon needle valve to allow the evacuation of the space ahead of the valve and the annular space. The main stem of the U tube was made up of 6 mm o.d. glass tube and the top bend section consisted of 2 mm bore capillary tube. The small cross section of the capillary bore ensured that the meniscus of the advancing mercury stream retained its hemispherical shape while flowing through the flat section of the bend.

A 180-mm-long, 0.25 mm i.d. capillary bore glass tube with a 0.2 mm o.d. tungsten wire was used at the end of the U tube to provide a high resistance to the flow of mercury. The capillary tube was joined to the U tube by sliding a plastic tube on the ends and sealed by using clamps. The use of the plastic tube gave some amount of flexibility to the joint which proved helpful during the operation of the apparatus. The lower end of the capillary tube was flared in order to accommodate a plastic pipet tube (PGC Scientific Corp., Gaithersburg, MD) that tapered down to an o.d. of 0.19 mm and an i.d. of 0.10 mm at the tip. The tip was held in place

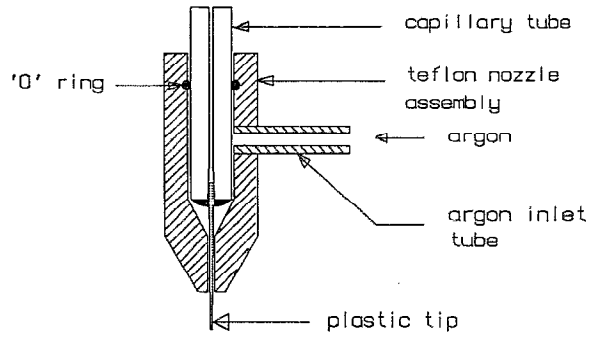

FIG. 6. The plastic tip/annular nozzle assembly.

with epoxy, which also formed a seal at the end of the capillary tube (Fig. 4).

An optical sensor (supplied by McMaster–Carr, Chicago, IL) was used to detect the growing drop. It was a reflection type of sensor and had a narrow (1.5 mm o.d.) fiberoptic head which consisted of two sets of fibers contained in a stainless steel sheath. One of these transmitted a narrow beam of light from the photosensor unit, which after reflection was picked up by the other set. The fiberoptic head was flexible and could be positioned close to the drop by moving the optical sensor, which was mounted on micrometer movement tables. Additional flexibility in alignment was provided by micrometer movements that made it possible to raise or lower the flask, which effectively raised or lowered the tip. The optical sensor was connected to a solenoid valve through a circuit (Fig. 5) designed to switch open the valve when the optical sensor detected the drop. This allowed for a pulse of argon to flow through the annular nozzle which forcibly detached the drop. The lower end of the nozzle had a bore whose diameter was slightly more than the o.d. of the plastic tip. The annular nozzle/capillary bore assembly with the tip is schematically shown in Fig. 6.

The gas jet used to detach the drop from the tip was switched on temporarily by a solenoid valve (SV1). The gas supply was connected to the valve through a dead volume (1000 cc capacity flask) to prevent a sharp reduction in the gas supply line pressure accompanying the outflow of gas in the jet. In the case of amalgam drop formation it was found necessary to shield the drop during its formation to minimize the loss of sodium due to reaction with oxygen/water vapor. This was done by providing a bypass in the gas supply line before the dead volume. A rotameter was used to regulate the flow rate in this bypass line.

The apparatus could be used to produce drops at a regular interval or to produce single individual drops whenever desired. To produce individual drops, a second solenoid valve (SV2) was connected to the mercury/amalgam reservoir, which was switched on immediately after drop formation to relieve the pressure on the metal pool and thereby stop the flow. The system could be repressurized by closing the valve (SV2) using a reset switch (described later), and this started the formation of a drop again. Drops of 0.6 mm diam and larger could be produced individually with ease by using this method. The smallest-sized drops (0.3 mm diam), could not be produced individually because of the inertia of the flow. This was because there was still a time lag, although very small, between the detachment of the drop from the tip and the lowering of pressure in the tapered flask. This meant that the flow of mercury/amalgam did not stop at the very instant the first drop was detached from the tip, which resulted in the formation of successive drops.

The entire apparatus was mounted on an aluminum base which was in turn placed in a tray (lined with felt) to collect mercury drops in case of a spill.

IV. OPERATION OF THE APPARATUS

Prior to the normal operation of the apparatus, it was connected to a vacuum unit which ensured that the metal filled the entire annular space formed by the tungsten wire in

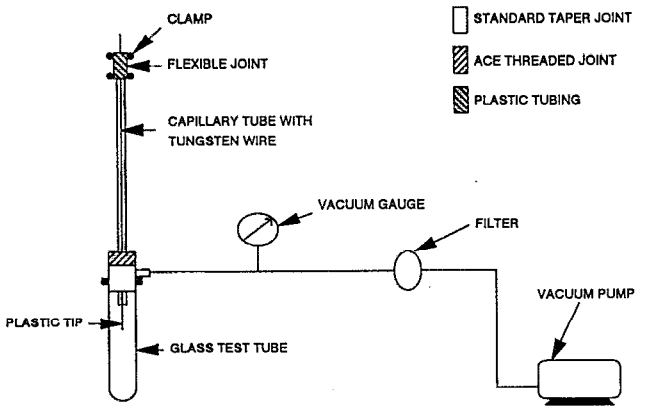

FIG. 7. Schematic diagram of the apparatus during the evacuation stage.

the capillary bore. As described earlier, this was done to ensure that no gas pockets were entrapped in the annular space, which would have induced instabilities during the growth of drops. The setup used during the evacuation stage is shown in Fig. 7. The vacuum level in the glass tube was held steady at 20 μmHg by the pump. From calculations,[8] it was estimated that approximately 17 h were required for the pump to bring the vacuum level in the space between the high vacuum stopcock and the capillary tube down to 100 μmHg, assuming no leaks.

To start the evacuation, the vacuum stopcock in the U tube was closed to prevent the metal from the reservoir from flowing into the space above the stopcock. After a couple of hours, the stopcock was opened partially to allow the metal to rise up to the stopcock level, slowly displacing all the air in the stem of the U tube below the Teflon stopcock. The stopcock was then closed with the mercury in the stem flush with the Teflon needle. After the evacuation period, the valve was opened again to allow the metal to flow into the U tube and then through the annulus, forming drops at the tip. The pump was allowed to run for some more time to make sure that the drop formation was regular.

After the evacuation stage, the vacuum unit was disconnected which stopped the formation of the drops at the tip. The annular nozzle was then assembled onto the capillary

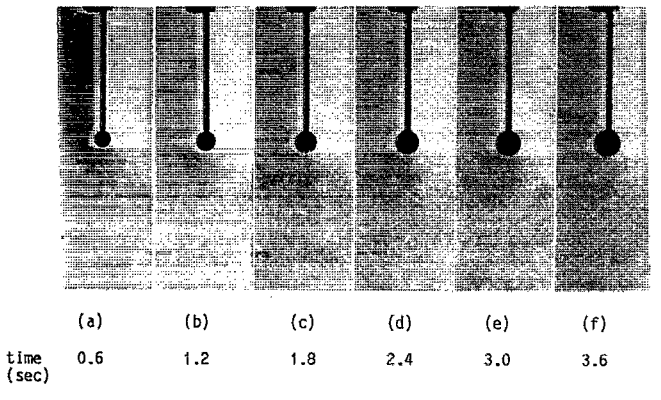

FIG. 8. The growth of an amalgam drop at the plastic tip. (0.6 s interval, o.d. of plastic tip=0.18 mm).

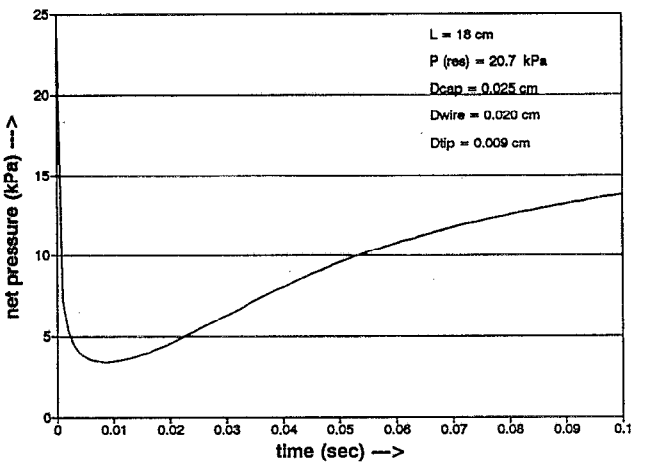

FIG. 9. The calculated ΔP–time relationship for P_{appl}=20.7 kPa.

tube making sure that the plastic tip did not bend in the process. This was done by sheathing the plastic tip with a stainless steel tube that had an i.d. larger than the plastic tube and an o.d. smaller than the bore diameter of the annular nozzle.

After ensuring that the annular nozzle was in place, and the tip was clean and dry, the argon gas supplies to the reservoir, the dead volume, and the first solenoid valve (SV1) were switched on. In the case of amalgam drops, the rotameter in the bypass line was regulated to maintain a very small flow rate to keep the drop flushed with argon. The pressures in both the supply lines were adjusted at the supply regulator to desired levels. Typical examples of the adjusted gauge pressures at the beginning were around 20.7 kPa (3 psi) over the reservoir and 34.5 kPa (5 psi) in the supply line to the dead volume. Usually the drop formation started at around 14 kPa (2 psi). The pressure over the reservoir was then adjusted to obtain the desired frequency of drop formation.

To get smaller drop sizes, the pressure in the reservoir was lowered to stop the formation of drops. The fiberoptic head of the optical sensor (described earlier) was aligned with the tip. In most cases it was necessary to adjust the

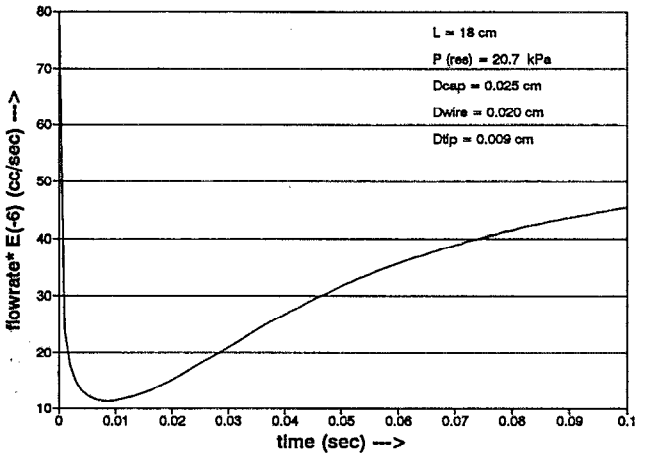

FIG. 10. The calculated flow rate–time relationship for P_{appl}=20.7 kPa.

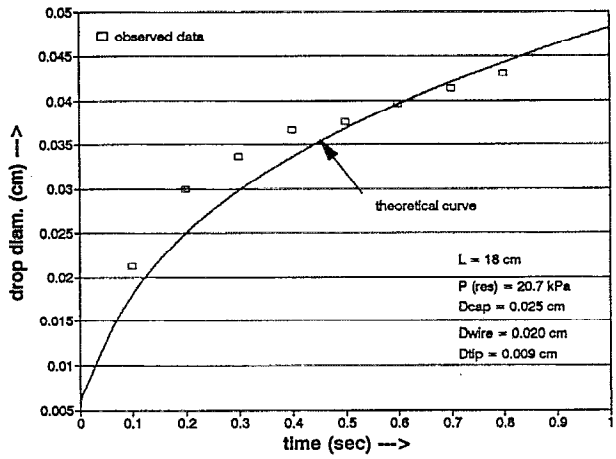

FIG. 11. Comparison of calculated and observed diameters of a growing drop for P_{appl}=20.7 kPa.

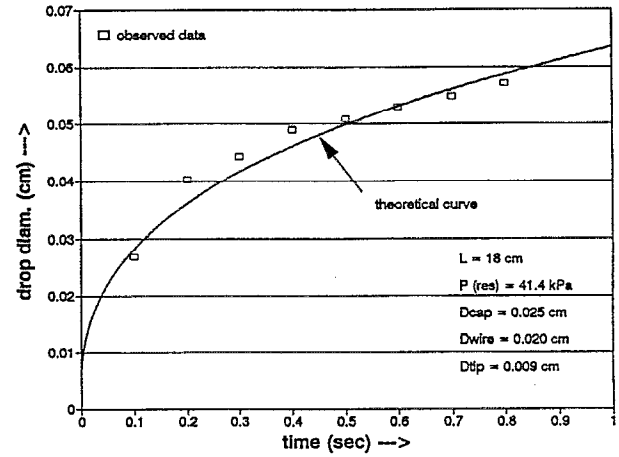

FIG. 12. Comparison of calculated and observed diameters of a growing drop for P_{appl}=41.4 kPa.

position of both the reservoir and the optical sensor, to bring the photosensor head in the same plane and at the same level as the plastic tip. Once they were at the same level the light-emitting diodes on the optical sensor would light up indicating that the photosensor unit had detected the light reflected from the tip. This was the reference height for the drop size and the sensor was lowered by a distance equal to the required drop diameter. However, to get better accuracy, this distance was later adjusted, after measuring the weights of drops to determine their sizes precisely.

As discussed earlier, the sensor was a reflective type and operated on a 12 V power supply. It put out a voltage equal to the supply voltage whenever the photosensor unit detected an object within its "sight." In our case the optical sensor was operated in the "light on" mode so that the output of the sensor was 12 V when the light reflected from the surface of

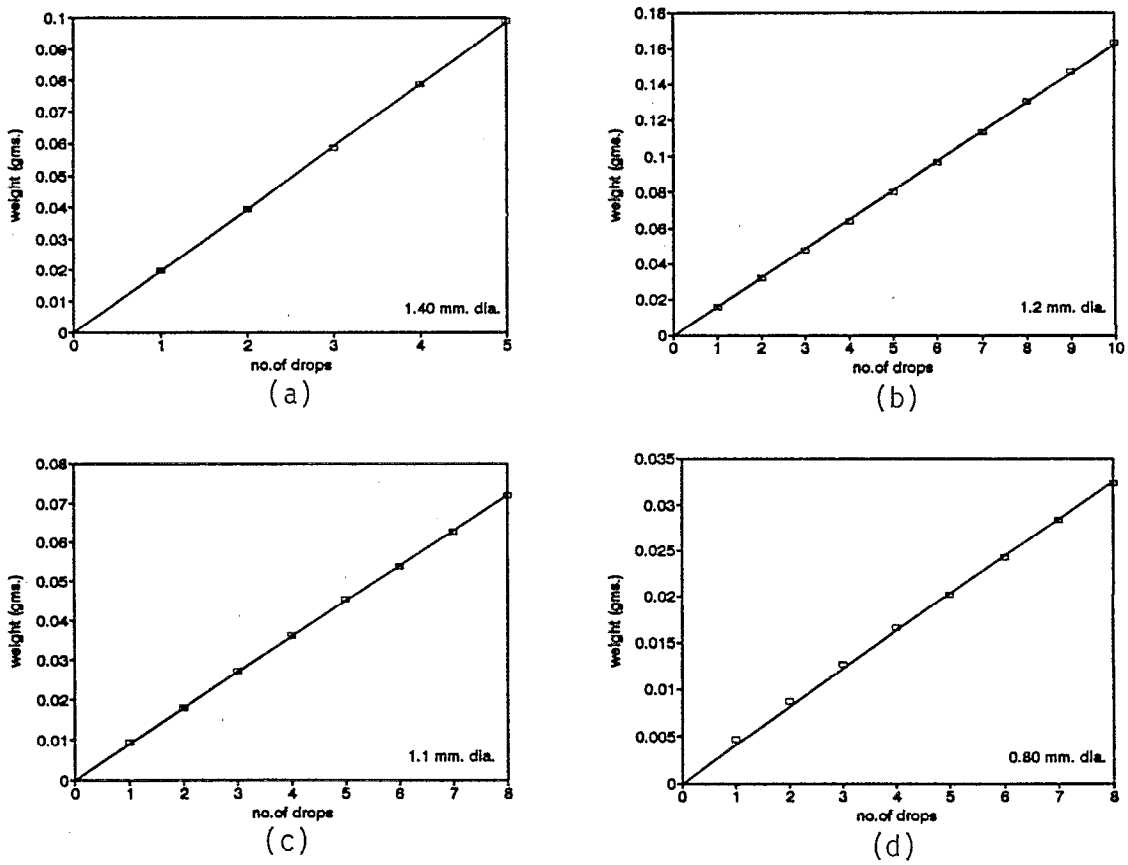

FIG. 13. Calibration curves for the apparatus obtained by measuring the weight of drops.

TABLE I. Measured drop diameters for two different sized drops.

1	0.745		1	0.356	
2	0.755		2	0.357	
3	0.755		3	0.356	
4	0.756		4	0.352	
5	0.761		5	0.356	
1	0.757		1	0.364	
2	0.746		2	0.379	
3	0.755		3	0.369	
4	0.754		4	0.369	
5	0.760		5	0.368	
1	0.753		1	0.360	
2	0.755		2	0.366	
3	0.760		3	0.364	
4	0.757		4	0.356	
5	0.748		5	0.363	
1	0.757		1	0.352	
2	0.765		2	0.360	
3	0.762		3	0.346	
4	0.762		4	0.349	
5	0.752		5	0.347	
1	0.753		1	0.352	
2	0.756		2	0.354	
3	0.751		3	0.346	
4	0.746		4	0.354	
5	0.762	avg. = 0.759	5	0.350	avg = 0.357
1	0.749	std = 0.007	1	0.352	std = 0.007
2	0.765	std(n-1) = 0.007	2	0.357	std(n-1) = 0.007
3	0.747		3	0.354	
4	0.751		4	0.353	
5	0.751		5	0.353	
1	0.761		1	0.352	
2	0.765		2	0.357	
3	0.764		3	0.355	
4	0.766		4	0.350	
5	0.769		5	0.355	
1	0.769		1	0.360	
2	0.765		2	0.363	
3	0.769		3	0.352	
4	0.772		4	0.356	
5	0.767		5	0.353	
1	0.765		1	0.364	
2	0.766		2	0.366	
3	0.768		3	0.364	
4	0.758		4	0.371	
5	0.761		5	0.359	
1	0.757		1	0.348	
2	0.759		2	0.350	
3	0.762		3	0.348	
4	0.763		4	0.351	
5	0.758		5	0.353	

the drop was detected by the photosensor. This output was then used to switch on the solenoid valves through a set of relays as shown in the circuit (Fig. 5). The output was first used to switch on relay No. 1 which then switched on the first solenoid valve (SV1). As a result of this, a jet of gas (argon) passed through the annular nozzle which detached the drop from the tip. As soon as the drop was detached the photosensor unit switched off and the relay returned to the normally open (NO) position. The second drop then grew until it reached the size when the photosensor unit detected it and the entire cycle would be repeated. To produce smaller drops, the photosensor unit was raised, reducing the vertical distance between the tip and the fiberoptic head, so that the drop was detected much earlier. Obviously, this increased the frequency of drop formation in addition to reducing the drop size. If it was necessary to reduce the drop formation frequency, the pressure in the reservoir was decreased which reduced the flow rate and slowed down the rate of growth of the drop. Conversely, the photosensor was lowered to get larger drops. This method produced a steady stream of drops of similar sizes. The typical period between drops was adjusted to 10–20 s.

The apparatus was also used to produce single individual drops. This was done by using the second relay (relay 2) in the circuit. With the switching of the first relay (relay 1) a voltage was impressed across SV2 and relay 2 at the same time, while the current flowed through the diode and switched on the relay. Once the relay was switched on, the circuit containing the reset switch was completed, and the relay stayed in the closed state (C). This also maintained a voltage across SV2 and so the solenoid valve remained open. The result of all this was that as soon as a drop was detected by the photosensor, all the relays and solenoid valves were switched on. With the detachment of the drop, relay 1 returned to the open position and SV1 closed, but relay 2 stayed closed (analogous to a latching relay) while SV2 remained open. Thus the pressure in the reservoir was released by the opening of SV2 and stayed the same way until the whole system was reset again. When it was time to form the next drop, the reset switch was used to temporarily break the circuit across SV2 which returned relay 2 to the normally open position (NO). The valve connected to the reservoir (SV2) closed and the pressure over the bath built up again to form another drop. To produce drops at a regular frequency the reset switch was set in the off position, breaking the circuit containing the second relay and SV2. This meant that the second relay never latched on with the break in the circuit at the reset switch. The diode in the circuit was used as a one-way switch to prevent the current from flowing back into the relay 1 circuit when relay 2 was in the latched mode.

V. RESULTS AND DISCUSSION

The instabilities that were observed due to entrapment of air in the initial designs of the experimental setups were completely eliminated by suitable design modifications and by filling up the annular space (with mercury) under vacuum. The growth of the drop at the tip was smooth as shown in Fig. 8, and the time required for the formation of same-sized drops at a constant applied pressure was the same from drop to drop.

The reliability and performance of the apparatus was judged by the measurement of various parameters, e.g., drop weight and size. The size of the growing drop was measured at various instants and then compared with the theoretical data to judge the performance of the apparatus. The reliability of the apparatus was measured by the consistency in the drop size from drop to drop at a particular setting.

The theoretical rate of growth of the drop was calculated using a program, which also calculated the net pressure difference (ΔP) and the effective flow rate (Q) at various instants.[8] Figures 9 and 10 show the pressure and flow rate, respectively, for an applied pressure of 20.7 kPa, as calculated by the program. These data were very useful in analyzing the different stages in the growth of the drop (described in an earlier section, Fig. 1). It was clear that the first stage of growth only lasted for a very short time (less than 1/100th s). The data for two different applied pressures showed similar trends.

Video recordings of the growing drop were analyzed using image analysis software to measure the growth rate of the drop.[9] Since the standard video recording is done at 30 frames/s, the smallest time interval of recording was 1/30th s. From this it was clear that the drop grew to a spherical cap larger than the hemispherical shape in a time far less than the time between frames in a video recording. Effectively the first picture in all the recordings showed a drop that was well into the third stage of its growth. For measurement purposes it was assumed that the first frame in the sequence corresponded to the size at 0.1 s from the start of the growth of the drop. The drop size in every third frame was measured and then the data were compared with the theoretically expected drop size, for two different applied pressures (Figs. 11 and 12). It was observed that the actual rate of growth during the initial period (of the third stage) was higher than the theoretically calculated values. However the trend in the growth rate was similar to the theoretical curve obtained from the program.

To meet the experimental requirements, it was essential that the drop sizes be consistent at a particular setting. As described earlier, the size of the drop could be controlled by raising or lowering the photosensor, thereby changing the vertical distance between the tip and the fiberoptic head. To get an idea of the reproducibility of the drop sizes produced by the apparatus, the relative position between the tip and the sensor was fixed and the size of the individual drops were then measured, using two different methods.

In the first method, the weights of individual drops were measured with a balance that had an accuracy of 0.1 mg, from which the size was calculated. Since the accuracy of this method was limited by the sensitivity of the balance, it was not suitable for smaller drops because of their extremely low weight. For example, a 0.80-mm-diam drop has a weight of 3.6 mg, which meant that an error of 0.1 mg (sensitivity of the balance) would have amounted to an error of 2.8% in the weight. In addition there was always the possibility of external errors which were much larger in comparison with the instrumental error, specifically in the case of low weights. Because of this, the method was limited to drops of 0.8 mm diam or larger. The calibration curves, in which the regression coefficient was found to be 0.98 or more for drops ranging from 0.8 to 1.4 mm in diameter, suggested a strong linear relationship between the number of drops and their weight (Fig. 13).

In the second method, the drops were allowed to pass through a viscous nonreactive fluid (silicone fluid—300 Cst). The passage of the drop was recorded at a high magnification (~100×) on a standard VHS video tape using an electronic CCD camera. The frames containing the image of the drops were analyzed using image analysis software to get the diameter of the drop. A comparison of the data obtained for two different drop sizes is shown in Table I. The tabulated data are for ten drops of each size, selected at random from a stream of drops, with five sets of measured diameters for each drop. The data show very good agreement with a coefficient of variation of 2%.

[1] S. M. Aeron, P. B. Crimes, and F. D. Richardson, Trans. Inst. Mining Metallurgy **83**, 167 (1974).
[2] W. R. Lane, J. Sci. Instrum. **24**, 98 (1947).
[3] N. R. Lindblad and J. M. Schneider, J. Sci. Instrum. **42**, 635 (1965).
[4] C. O. Pedersen, Int. J. Heat Mass Transfer **13**, 369 (1970).
[5] R. L. Verma, R. C. Bapna, and D. N. Joshi, J. Sci. Instrum. **22**, 189 (1989).
[6] R. B. Bird, W. E. Stewart, and E. N. Lightfoot, *Transport Phenomena* (Wiley, New York, 1960), p. 51.
[7] L. Liggieri, F. Ravera, and A. Passerone, J. Colloid Interface Sci. **140**, 436 (1990).
[8] D. Panda, M. S. thesis, Metallurgical Engineering, University of Missouri-Rolla, MO, 1993.
[9] K. Peaslee, Semi-Annual Report No. 9, Generic Mineral Technology Center for Pyrometallurgy, UMR, Rolla, MO, October 1991–March 1992, p. 31.