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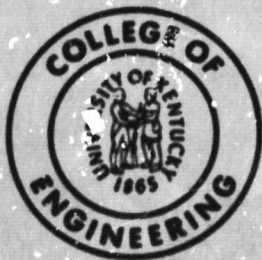
DYNAMICS AND STABILITY OF ELECTROLYTIC BUBBLES: BUBBLE DEPARTURE DIAMETERS

by

GAUTAM TRIVEDI

and

JAMES E. FUNK

Boiling and Phase-Change Laboratory
Department of Mechanical Engineering

OFFICE OF RESEARCH AND ENGINEERING SERVICES

COLLEGE OF ENGINEERING
UNIVERSITY OF KENTUCKY

Sgt-64864R

Technical Report UKY 28-70-ME 8

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Gautam Trivedi, Research Assistant

James E. Funk, Professor of Mechanical Engineering

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Department of Mechanical Engineering

University of Kentucky

1970

PREFACE

This report describes the initial findings of the Bubble Dynamics Project, which is part of a larger Themis Project entitled <u>Studies of Electrochemical Processes</u>, Grant DAA B07-69-C-0366. This work is part of a joint effort with NASA in which we are attempting to establish the similarity between the processes of boiling and electrolysis so that work done in one area may be made useful in the other. The joint effort is supported by NASA Grant NGR/18-001-035, Gravity Boiling Project, under the direction of Professor John H. Lienhard.

We are grateful to Professor Lienhard and to Mr. Amit Bhattacharya for their help with this report.

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I. INTRODUCTION

During electrolysis, the evolution of gas bubbles at the electrolyte-electrode interface consists of a series of consecutive steps. After the charge transfer reaction and subsequent formation of the product molecular species, the gas dissolves in the liquid and begins to diffuse away from the electrode surface. Due to the slowness of the diffusion process, the liquid soon becomes sufficiently supersaturated for nucleation to begin spontaneously. The bubble grows with supersaturation serving as the driving force until finally the buoyancy forces overcome the surface forces and separation occurs.

Using the results of Bashforth and Adams [1] and Wark [2], Fritz [3] obtained an expression for the maximum volume of a vapor bubble which can remain attached to a solid surface. His expression made use of the assumption that the buoyancy just balances the surface tension force.

Under the additional assumption that the bubbles are small and nearly spherical, the small volume due to contact with the solid surface can be neglected compared to the total bubble volume, and the Fritz equation for the bubble departure diameter, D_{max}, obtained as

$$D_{max} = 1.184\theta \left(\frac{\sigma}{g(\rho_l - \rho_g)} \right)^{1/2} \qquad (1)$$

where θ = contact angle in radians

σ = surface tension

ρ_l = density of the liquid

ρ_g = density of the gas

This equation was verified experimentally in numerous examples of nucleate boiling by Han and Griffith [4].

Coehen and Neumann [5] studied experimentally the sizes of bubbles on horizontal electrodes for several different electrolytes and under different current densities. It was shown that in alkaline solutions smaller bubbles separate from the cathode but large ones from the anode, and vice-versa in acidic solutions. Also, bubble size was reduced with increasing dilution of the electrolyte or with increasing current densities. They postulated an electrostatic theory to explain this phenomenon. On the basis of their theory, bubbles were taken to be charged and could therefore interact with the electrode. An electrostatic attraction would have the effect of increasing the surface forces and, consequently, the bubble detachment diameter would increase while an electrostatic repulsion would have the opposite effect and lead to the formation of smaller bubbles. In order to explain the effects of the electrolyte, the charge on the bubbles must be a function of the concentration of the solution and the nature of the electrolyte. Coehen and Neumann postulated an explanation but made no attempt to give a quantitative explanation.

Kabonov and Frumkin [6], using data on the mobility of bubbles in electric fields, found estimates of the electrical forces and compared them to the bouyancy forces. For the size of bubbles observed by Coehen and Neumann, the electrical forces were found to be at least six orders of magnitude less than the bouyancy forces. Thus, obviously, electrostatic attraction or repulsion in this manner cannot account for the experimental evidence and alternative explanations are necessary.

It was shown by Möller [7] that the contact angle of gas bubbles depends on the potential of the solution. This dependence is very similar to the electro-capillary curves of mercury. The parabolic nature of the contact angle-potential curve was confirmed by Frumkin and co-workers [6,7,8,9,10]. Equation (1) gives the relationship between the contact angle and the detachment diameter. There is not, however, enough quantitative data on the contact angle-potential dependence to calculate the maximum size of bubbles in the experiments of Coehen and Neumann. It has been mentioned above that the effect of charge on bubbles is not important in the force balance. There is, however, the possibility that the contact angle has a dependence on the charge. If, in fact, there is such a dependence it should have an effect on the stability of the bubble. There is evidence [10] that the surface charge does affect the contact angle but no quantitative information is as yet available.

Attempts have been made by McTaggart [11], Alty [12], and Bach and Gilman [13] to find the charge on bubbles. However,

due to experimental uncertainties and the difficulties in accounting for electro-osmotic effects, only the sign of the charge has been ascertained. The effect of this charge on the bubble interactions is still generally unknown.

There is a need for studies of the nucleation, growth and separation of electrolytic gas bubbles from solid electrode surfaces, to evaluate existing theories as has been discussed recently by Cheh [14], and a need for new reliable theories as pointed out by Bhattacharya and Lienhard [15]. The work described in this report is concerned with obtaining and interpreting bubble contact angles and bubble detachment diameters. No attempts have been made in the literature to explain analytically the experimental results of Coehen and Neumann, mainly because of lack of contact angle data for electrolytic gas bubbles growing on horizontal electrode surfaces. Cohen and Neumann obtained their data for bubbles growing on a horizontal electrode by photographing the electrode surface through a microscope looking vertically downwards. As a result, the maximum bubble diameter could be obtained but no contact angle measurements were possible. It is the object of this work to devise a method for growing gas bubbles (H_2 and O_2) on solid horizontal electrode surfaces (Platinum) and measuring the contact angles and the departure diameters. These data may then be used to check the validity of Equation (1), or if necessary to develop a correlation for the maximum bubble diameter and the contact angle.

II. EXPERIMENTAL APPARATUS AND PROCEDURE

Since this study is concerned with microscopic details, small electrode dimensions were necessary. Two different

electrodes were used, one made of ⌐ .002 inch diameter platinum
wire and the other of .004 inch thick platinum foil. The electrode
was attached to an L-shaped plastic support with epoxy cement
as shown in Figure 1(a). The electrical lead was taken out
through a hole in the side of the plastic test cell, which was
sealed with a silastic sealant. The anode was a #10 Nichrome
wire, with its end-face polished, sealed into the opposite face
of the test cell, Figure 1(b).

To obtain greater clarity in the field of view and maxi-
mum brightness, the two longitudinal sides of the test cell
were made of thin glass plate cemented to the plastic with
silastic. This cell was mounted on a microscope laid on its
side so that the objective was horizontal, Figure 2. In this
position the microscope could be focused on the horizontal
electrode surface through the glass side. The difficulty in
focusing, due to the small working distances involved in most
high magnification objective lenses, was avoided by the L-shape
of the electrode holder, which brought the electrode surface
sufficiently near the glass wall.

The polishing of the cathode surface (wire tip or foil
edge)was carried out with five grades of emery cloth, the last
being 4/0. The last stage used a power driven rotary disc
covered with canvas and wetted with alumina which was fine
enough to remain suspended in water for 10 minutes. Between
stages, the cathode surface was rinsed in alcohol, dried and
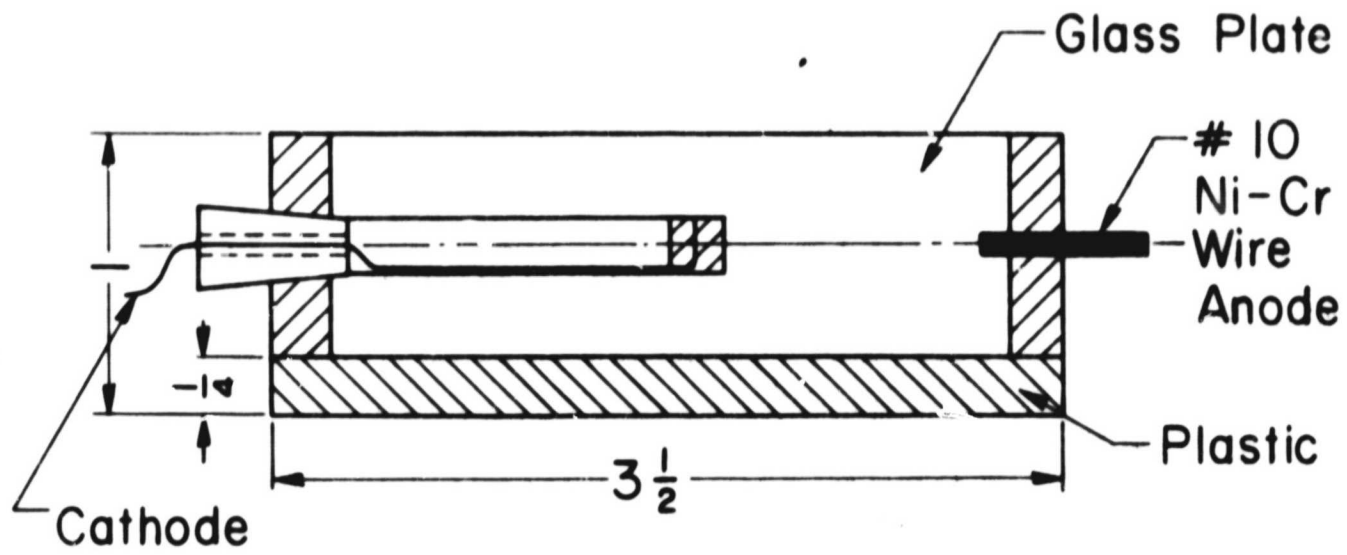turned 90°.

Glass Plate

10
Ni-Cr
Wire
Anode

Plastic

$3\frac{1}{2}$

Cathode

Figure I(a). Elevation

Glass Plates
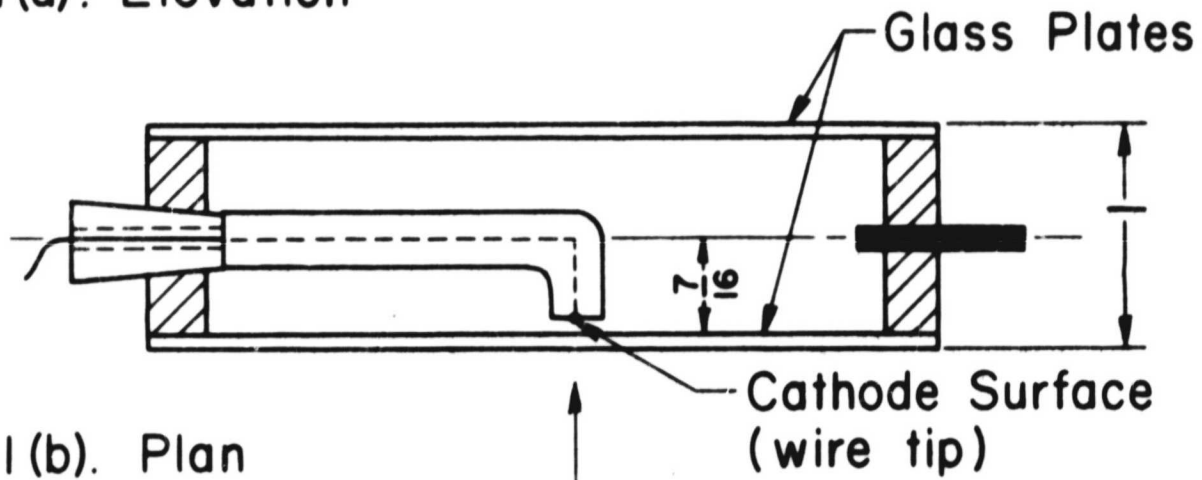
$\frac{7}{16}$

Cathode Surface
(wire tip)

Figure I(b). Plan

Microscope
Axis
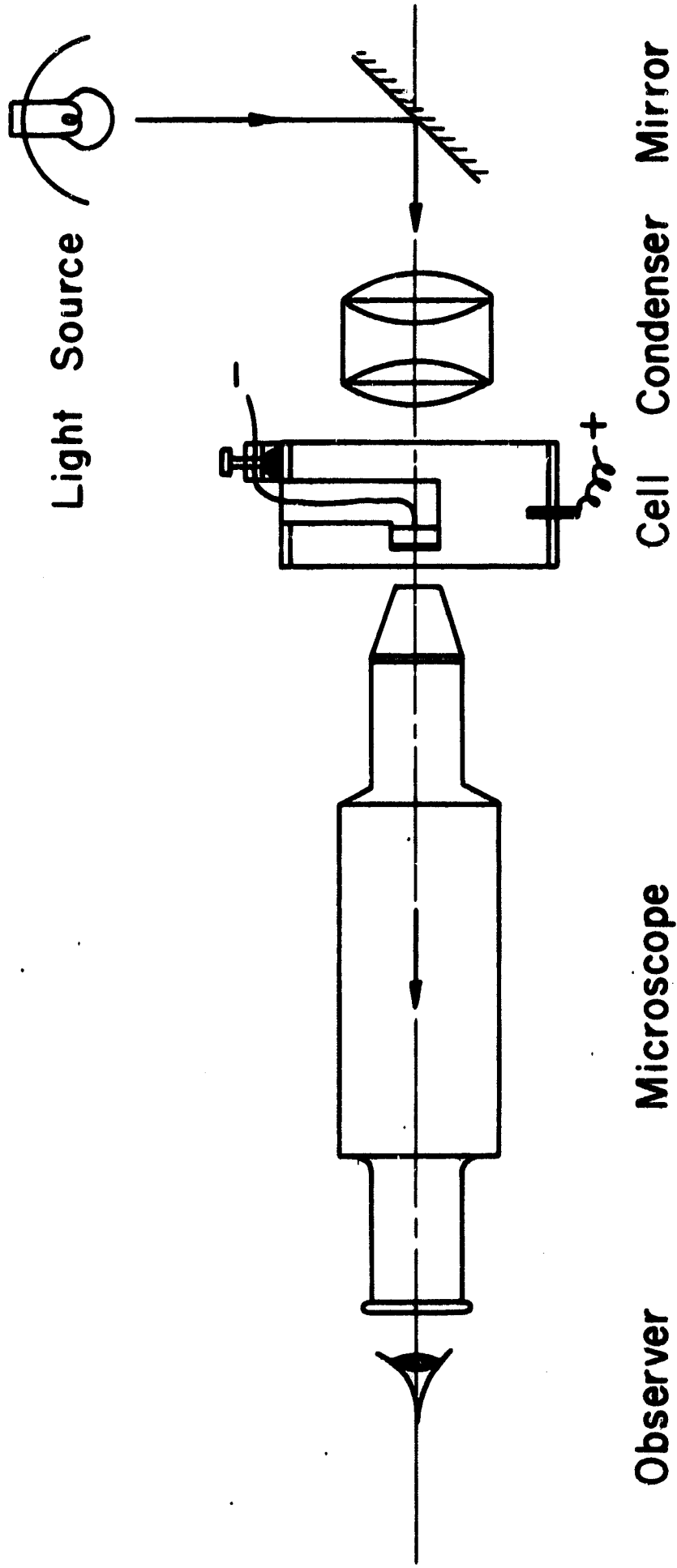
Figure 1. Schematic diagram of the test cell.

Figure 2. Plan view of the optical arrangement.

The electrolyte was 1.0 N solution of KOH. The cell
contained about 60 cc of the test liquid during one run.

The electrical circuit was as shown in Figure 3, and
the voltages and currents were controlled by suitable voltage
dividers and resistances and recorded by a voltmeter and a
microammeter. The voltage applied to the test cell during
any one run was kept constant.

The microscope gave a total magnification of 43.5 and
the grid in the eyepiece was found to be .001 x .001 inch on
calibration. For ordinary work, a 150W-110V lamp light
source was used while for photography, a quartz flood lamp
[spec. 650W-120W] was focused through the microscope con-
denser lense onto the electrode surface.

When it was desired to photograph the electrode surface,
a Nikon [Model Nikkormat, using a 50mm, f/2 lense] camera was
focused through the microscope eyepiece, with a black cardboard
tube acting as a light shield, and the film exposed at the
desired time while viewing through the reflex lens. The elec-
trolytes used in the experiments were first prepared in double
distilled water in the required concentrations. The test cell
was washed with distilled water and then rinsed out with the
electrolyte to be used for that run. Prior to each run, the
microscope was relevelled and the test cell clamped in place so
that the electrode was horizontal. The electrolyte was trans-
ferred to the cell using a similarly cleaned pipette. With
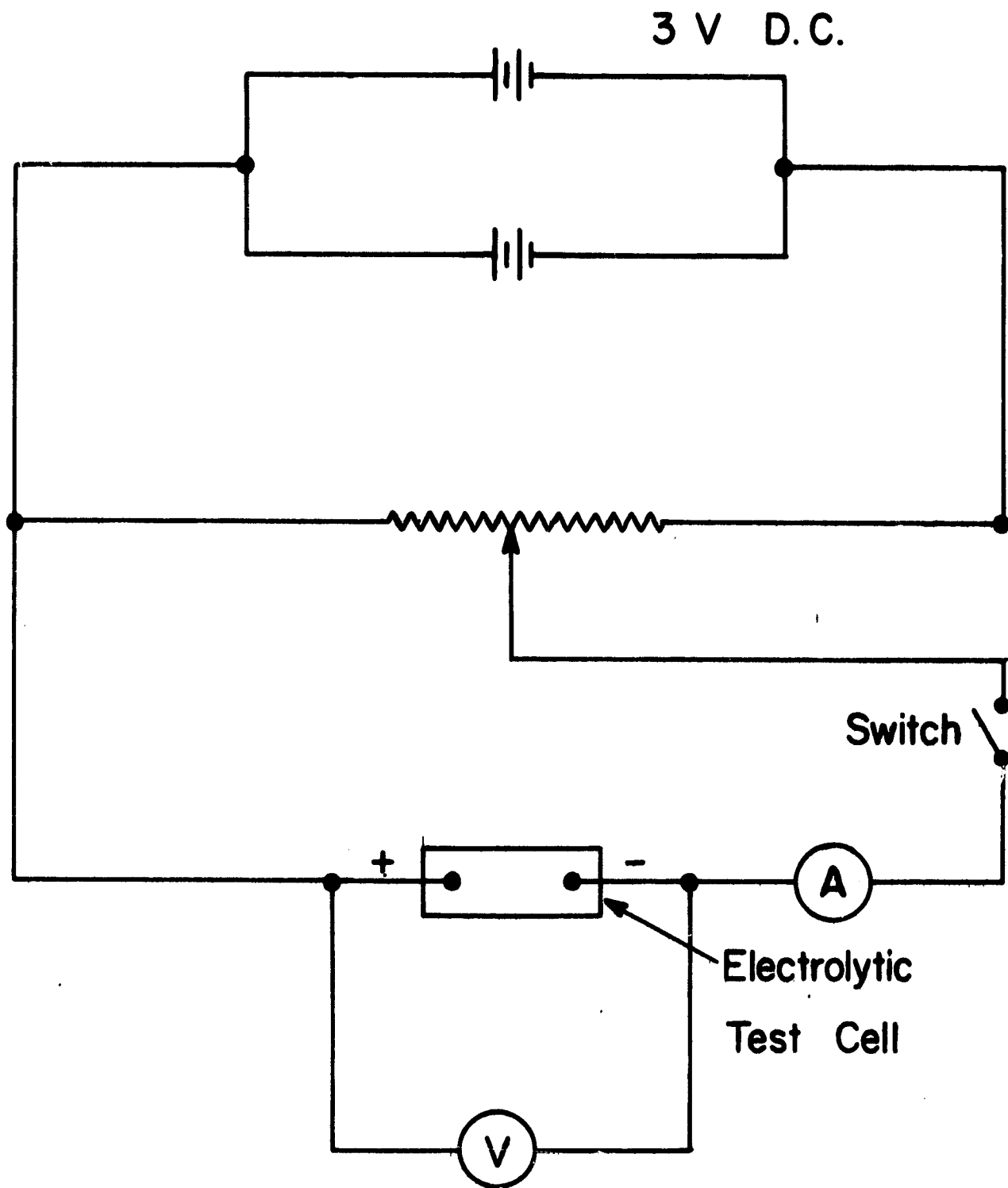
Figure 3. Electrical circuit used to grow the electrolysis bubbles.

the electrical connections in place, the microscope was focused on to the electrode surface.

When it was desired to observe continuous growth of the bubbles, a toggle switch was used in the circuit and when a particular bubble at various stages of growth was to be studied, a telegraphic key type of switch was used.

Since the bubbles are not all of the same size as they detach from the electrode, it was found necessary to compile data on the bubble departure diameter for a particular concentration and voltage. In this way, the effects of concentration and voltage on bubble size could be found and contact angle data determined accordingly. For the statistical study of the bubble departure size, the electrode was scanned from one end to the other by traversing the microscope and the bubble size for a particular voltage and current read directly from the microscope grid. As many as ten to fifteen consecutive bubbles were observed from each site and all the active sites on th electrode were studied.

Enlargements were made from photographs of bubbles on the point of departure such that the overall magnification was as much as four hundred times. The contact angles and the radii were then measured for use with Equation (1).

According to Westwater [16], bubble growth character-istics are essentially the same when the cell is operated at constant voltage as when it is operated at constant current.

All the data presented in this report were obtained at constant voltage.

The preliminary investigations have been done mainly on 1.0 N KOH solution although the behavior of other acid and base electrolytes is under study as the experimental arrangement has been found to be satisfactory.
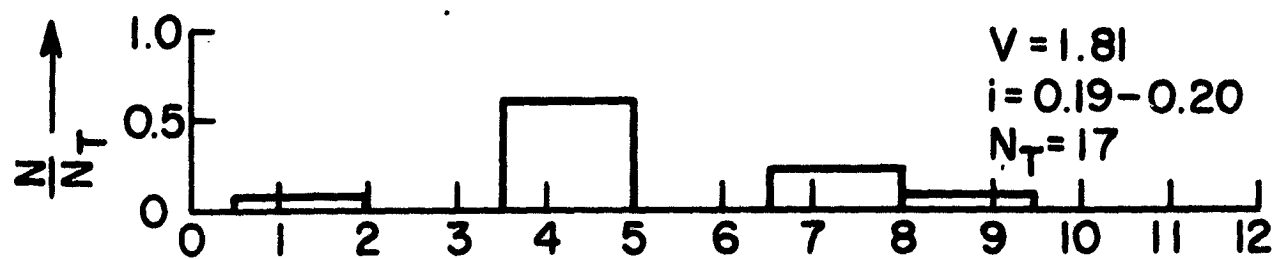
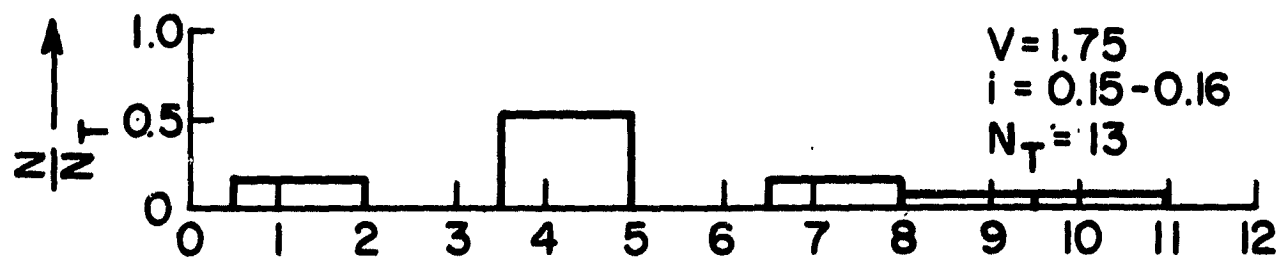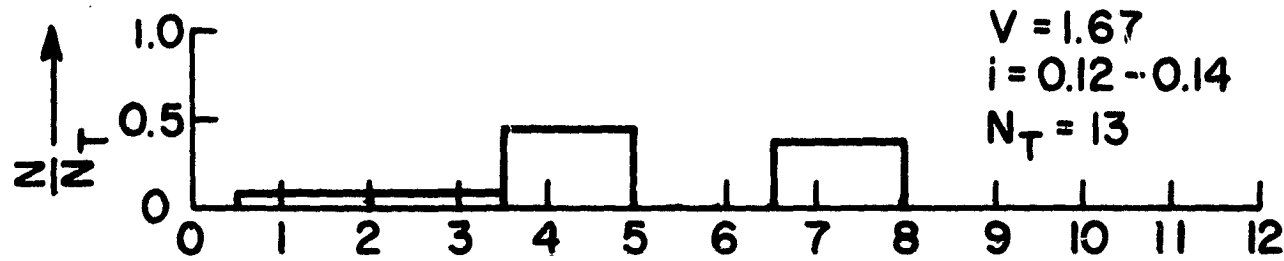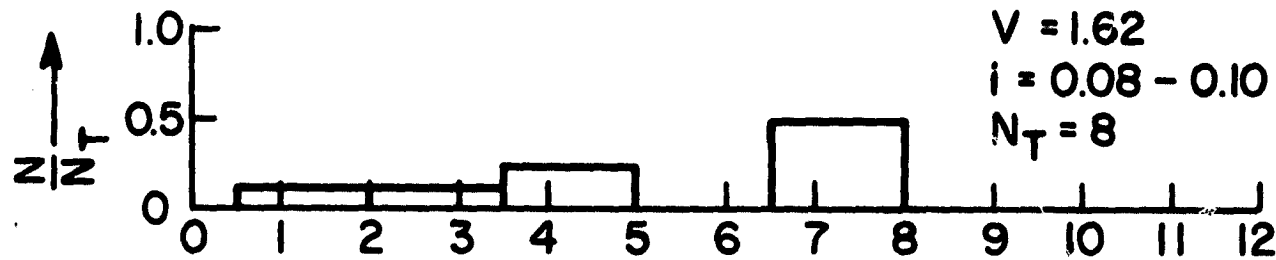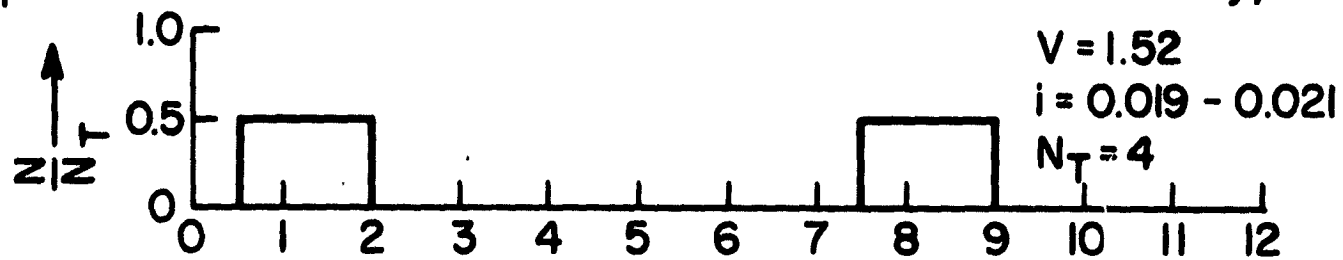## III.   RESULTS AND DISCUSSION

A.   Bubble Size Distribution

The data on the bubble departure diameter for hydrogen at different voltages has been plotted in Figure 4.   The bubbles were generated in a 1.0 N KOH solution and grew on the horizontal electrode surface.

It can be seen that as the voltage (or current density) increases, the fraction of sites producing bubbles in the range 3.5 to 5.0×10^{-3} inches increases sharply.   In general, there are two distinct types of bubbles, one that grows to a fairly large size (7.0 to 30.0×10^{-3} inches) before leaving the electrode surface and another that departs at a relatively smaller size (0.5 to 5.0×10^{-3} inches).   As the rate of gas evolution increases, more bubbles of the smaller size are produced.

We found that between the departure of one bubble and the nucleation of the next from a particular site there was a certain fixed time.   This waiting time was found larger for the larger bubbles than for those that grew to a smaller size. As a result the large bubbles were seen to grow individually whereas the small ones appeared as a continuous stream.

N — Number of Active Sites   V — Voltage , Volts
N_T — Total Number of Active Sites   i — Current Density, ma/in²

Figure 4.   Hydrogen bubble departure diameters for various current
densities in 1.0 N KOH solution.

For nucleate boiling in the discrete bubble region, Cochran, et. al. [17, 18, 19] observed that there was evidence of flow around the liquid-vapor interface both during bubble growth and collapse. It may be conjectured that a large number of small bubbles are formed at the higher current densities partly due to the forces associated with the momentum of the liquid surrounding the bubbles. This force has also been termed the inertia force by other workers and may be responsible for causing bubbles to break off at smaller than normal diameters.

The mechanism by which the momentum of the liquid affects the bubbles has not been determined exactly. Han and Griffith [4] suggest that the momentum imparted to the liquid ahead of the bubble tends to lift the bubble off the surface if the growth of the bubble decelerates rapidly enough. For such bubbles, the inertial force alone rises fast enough to overcome the surface forces. The buoyancy force at this stage is still quite small and does not significantly affect the bubble dynamics. According to Siegel [20], if a particular nucleation site emitted rapidly growing bubbles, then these would most likely exhibit a more significant inertial effect.

Schrock, et. al. [21] have recently obtained a solution for the flow field in a semi-infinite fluid surrounding a sphere that is growing while tangent to a plane solid surface. Using these results, Mikic ,et. al. [22] have derived a general relation for bubble growth in a uniformly superheated liquid and in a non-uniform temperature field. This relation is valid

in both the inertia controlled and the heat diffusion controlled
growth regions, and agrees well with nucleate boiling data.
They also found that a waiting time of the same order of magni-
tude as the departure time reduced the bubble radius almost by
a factor of two.

Although references [4], [17], [18], [19], [20], [21]
and [22] referred to nucleate boiling bubbles, it has been
found in the course of this study that similar phenomenon
occur during electrolytic gas evolution and may have a similar
explanation. Thus, we suspect that inertial forces play an
important role in the detachment of the smaller sized bubbles
with buoyancy effects being small; while, for the larger sized
bubbles, buoyancy is an important factor in determining the
bubble detachment diameter. The waiting time could also be an
important factor in determining the departure diameter.

B. Contact Angle-Diameter Data

The visual and photographic observations reveal that
the hydrogen gas bubbles formed on the horizontal surface are
very nearly spherical in shape. The angle of contact was
found to be small, between 15 to 35 degrees. The bubbles were
found to be almost spherical even at early stages of growth
and remained nearly so throughout their period of attachment
to the electrode surface.

In view of the fact that the bubbles in electrolysis were
found to be nearly spherical, it was decided to compare
the results of Equation (1) with an analysis in which the bubble
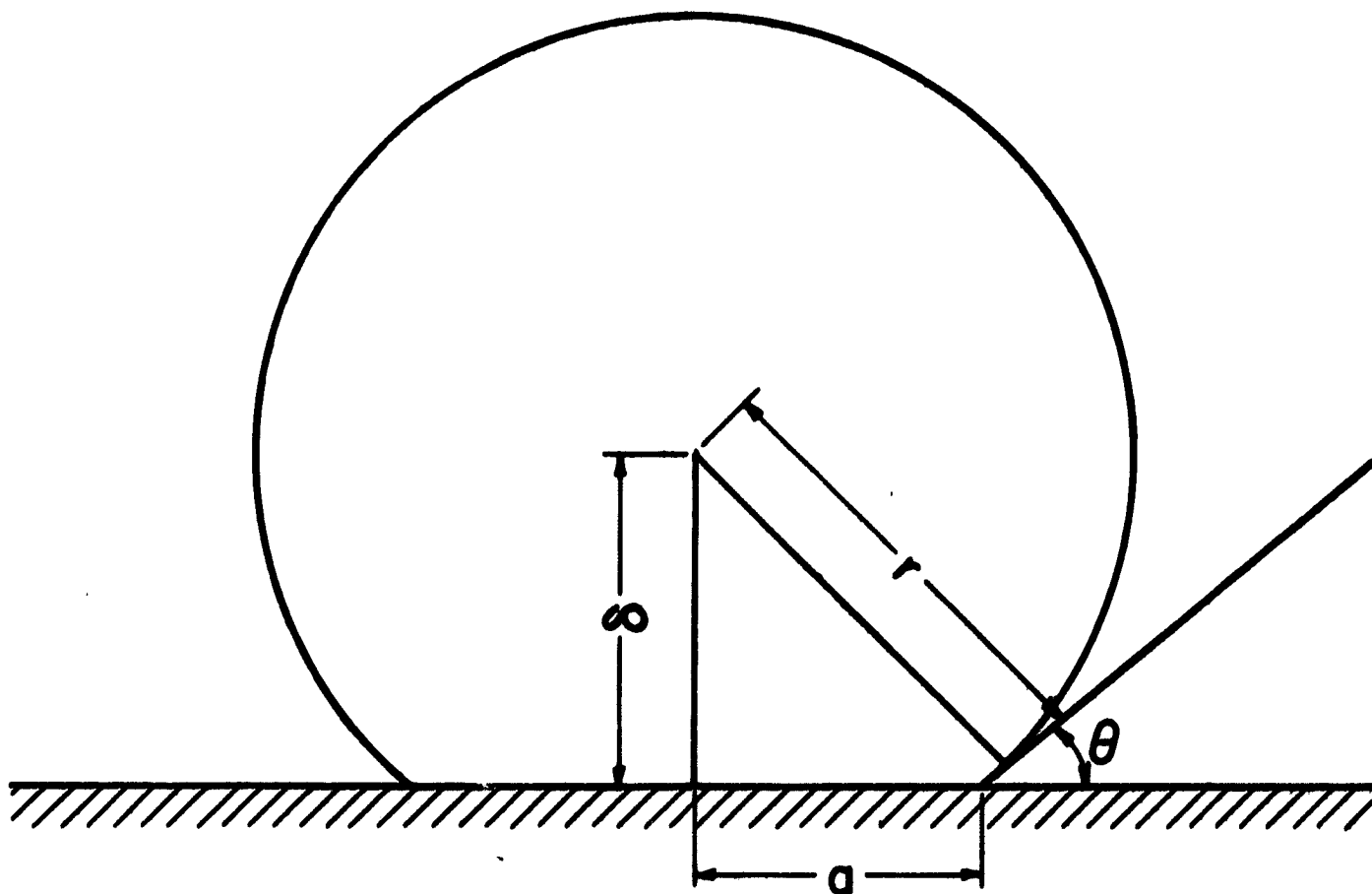is considered to be part of a sphere, as shown in Figure 5. The

Figure 5.   Schematic diagram of bubble as part of a sphere.

volume, V_{gas}, of the bubble on the electrode can be written as

$$V_{gas} = \frac{4}{3} \pi r^3 - \frac{1}{3} \pi (r-\delta)(3r - \{r-\delta\})$$

$$= r^3 \left[\frac{4}{3} \pi - \frac{\pi}{3} \left\{ 2 - 3 \cos \theta + \cos^3 \theta \right\} \right] \qquad (2)$$

where r, δ and θ are shown in Figure 5 and

$$\cos \theta = \frac{\delta}{r}$$

Also, equating the bouyancy and surface tension forces for the part of the sphere shown, we get

$$V_{gas} \left(\rho_\ell - \rho_v \right) g = 2\pi \alpha \sigma \sin \beta$$

$$= 2\pi \sigma \frac{\alpha^2}{r}$$

$$\text{since } \beta = \frac{\alpha}{r}$$

$$V_{gas} = \frac{2\pi \sigma (r^2 - \delta^2)}{g(\rho_\ell - \rho_v) r}$$

or
$$V_{gas} = \frac{2\pi r \sigma (1 - \cos^2 \theta)}{g(\rho_\ell - \rho_v)} \qquad (3)$$

Neglecting the density of the vapor, ρ_v, in comparison to the density of the liquid, ρ_ℓ, and equating (2) and (3), we get

$$D_{max} = \left(\frac{24 \sigma (1 - \cos^2 \theta)}{g \rho [2 + 3 \cos \theta - \cos^3 \theta]} \right)^{1/2} \qquad (4)$$

Equations (1) and (4) have been plotted in Figure 6 for different values of the contact angle. The latter gives larger values of the departure diameter than the Fritz formula (Equation (1)) which has been verified for nucleate boiling by Han and
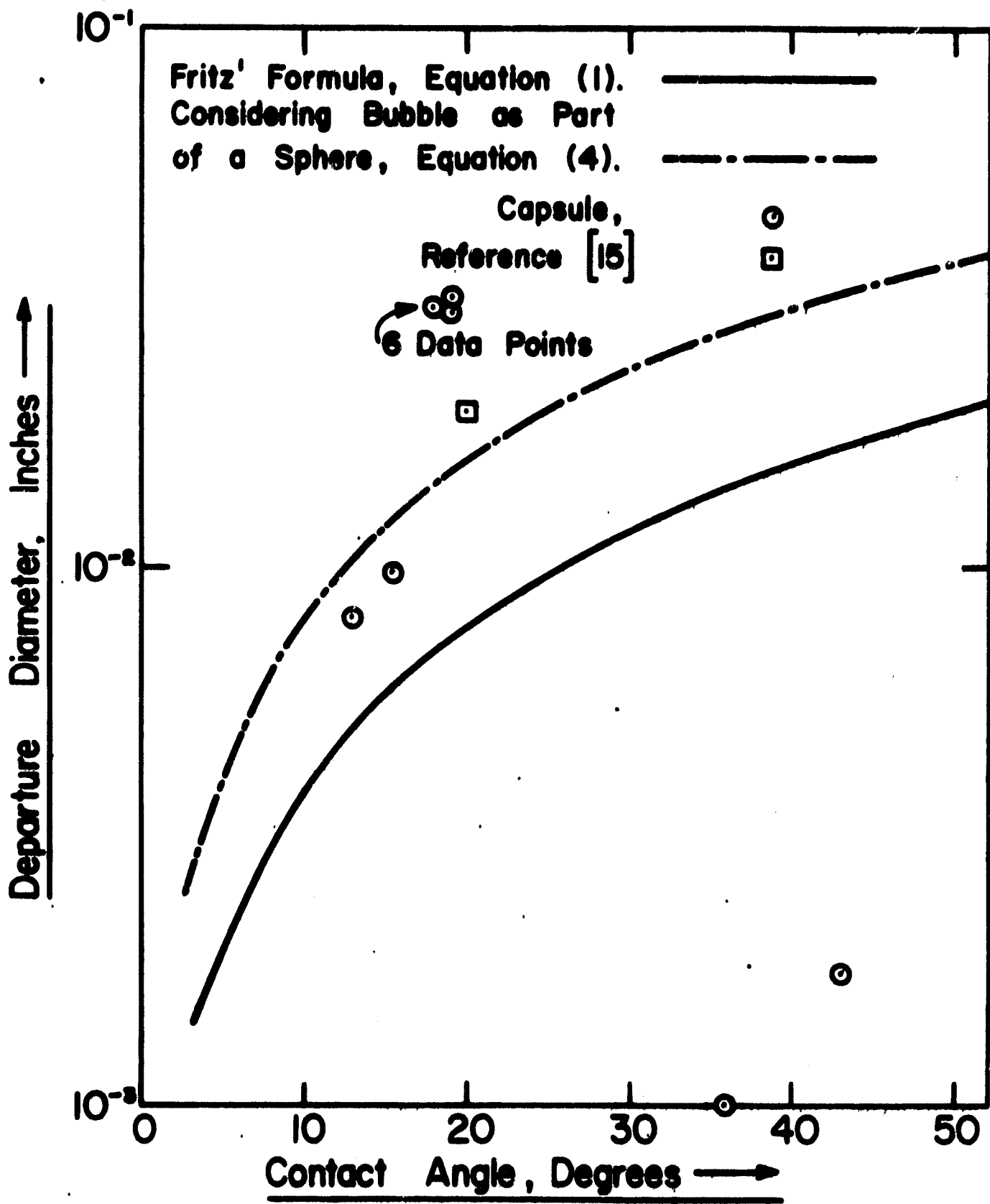
Figure 6. Hydrogen bubbles on a horizontal surface--bubble departure diameter versus contact angle in 1.0 N KOH solution.
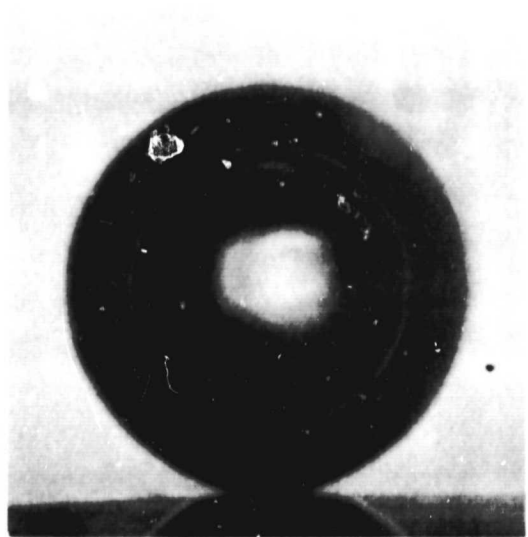
Griffith [4]. The value of the surface tension, σ, and the density of the liquid, ρ_ℓ, in Equations (1) and (4) were taken to be the standard values given in reference [23]. The values used were as follows:
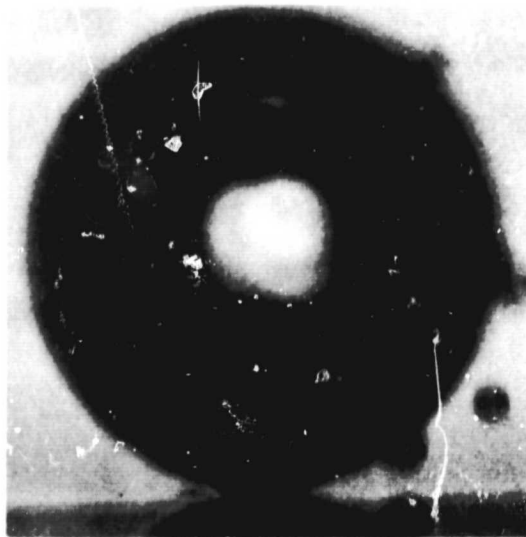
Surface tension, $\sigma = 0.005135$ lb$_f$/ft.

Density of 1.0 N KOH $\rho_\ell = 65.6014$ lbs/ft^3

Data obtained for the bubble departure diameter and contact angle have also been shown in Figure 6. It is found that the very small bubbles, in the range .5 to 2×10^{-3} inches fall far away from either curve and may be said to be independent of the gravity effect (bouyancy force). On the other hand, the larger bubbles have been found to be larger than those predicted by Fritz' formula and by Equation (4), and may be dependent to a large extent on the bouyancy effect for detachment. However, since this alone underestimates the size of the bubble, the presence of other effects like the effect of charge on contact angle tending to hold the bubble may be conjectured. No conclusions about the nature of these may be drawn at this stage and the need for further investigation with other electrolytes is felt necessary.

Figures 7 and 8 are some examples of the photographs obtained for the contact angle-detachment diameter data. Enlargements of the pictures were made to facilitate the measurement of the contact angle and the diameter, the microscope grid being used for calibration. These photographs were made looking through the microscope at the base of the hydrogen bubbles growing on the horizontal electrode. The bubbles from a site were first observed to find out the detachment diameters and then when the next bubble
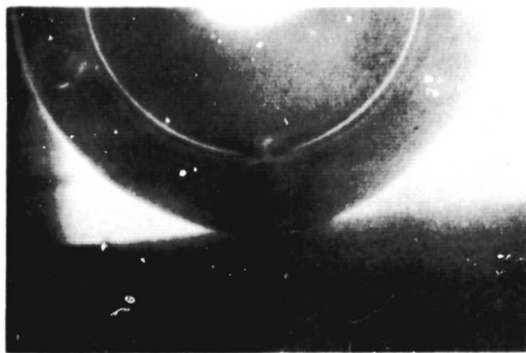
(a) Bubble diameter= 0.008 in.
Contact angle: left= 10°
                  right= 16°
Voltage= 1.81 V

(b) Bubble diameter= 0.0095 in.
Contact angle: left= 16°
                  right= 15°
Voltage= 1.81 V
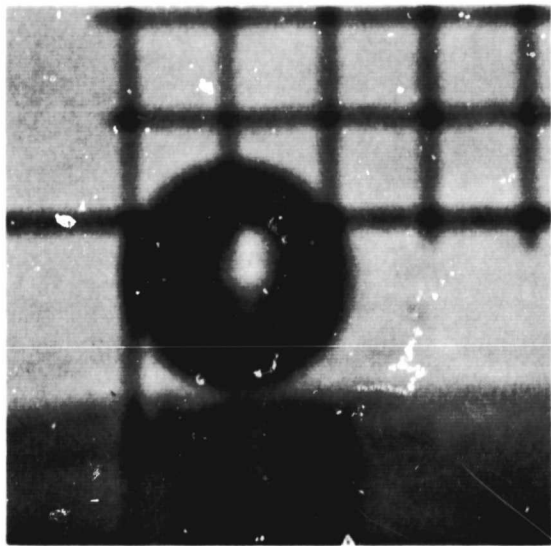
(c) Bubble diameter= 0.031 in.
Contact angle: left= 14°
                  right= 18°
Voltage= 1.58 V

(d) Bubble diameter= 0.030 in.
Contact angle: left= 18°
                  right= 18°
Voltage= 1.58 V

Figure 7.   Hydrogen bubbles in 1.0 N KOH solution on the foil edge
            electrode.

(a) Bubble diameter= 0.002 in.   (b) Bubble diameter= 0.0015 in.
    Contact angle: left= 36°   Contact angle: left= 38°
                  right= 28°   right= 36°
    Voltage= 1.57 V   Voltage= 1.57 V

Figure 8.   Hydrogen bubbles in 2.14 N KOH solution on the wire tip
           electrode.

reached that particular size, the circuit was switched off and the photographs taken. To confirm that the bubble really was on the point of departure, the process was carried on and if the bubble did not depart immediately, more photographs were taken as felt necessary.

All the experiments were ran at 70°F and at atmospheric pressure. Figure 7(a), (b), (c), and (d) show hydrogen bubbles growing on a horizontal 0.004 x 0.25 inch platinum foil edge, while Figure 8(a) and (b) show hydrogen bubbles growing on a 0.002 inch diameter horizontal platinum wire tip.

The contact diameter at the base of the bubbles is small compared to the size of the bubbles. As Figures 7 and 8 show, the contact diameter is of the order of 0.001 to 0.002 inch for bubbles in the range of 0.005 to 0.010 inch, and remains of the same order for even the largest bubbles.

C. Other Photographic Observations

During the course of the present investigations, some interesting examples of bubble interaction were noticed. It was found that when there were two sites very near each other, they continuously produced bubbles of a small size which departed on contact. However, after a certain time, due to differences in waiting time, one would lag the other by a time that was sufficient to let the first bubble grow to a larger size. Under these circumstances, the second site would continously produce small bubbles (0.0005 to 0.001 inch) that coalesced with the larger bubble (see Figure 9(a)) until it was large enough to

(a) Bubble Diameter= 0.025 in.
    Contact Angle: left= 19°
                    right= 26°

    Voltage= 1.58 V

(b) Bubble diameter= 0.024 in.
    Contact Angle: left= 18°
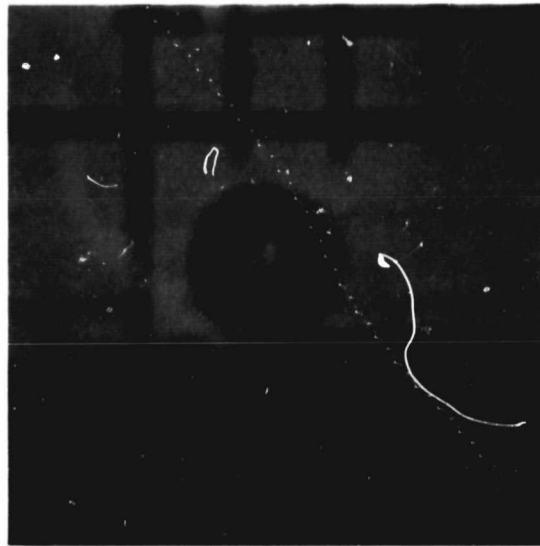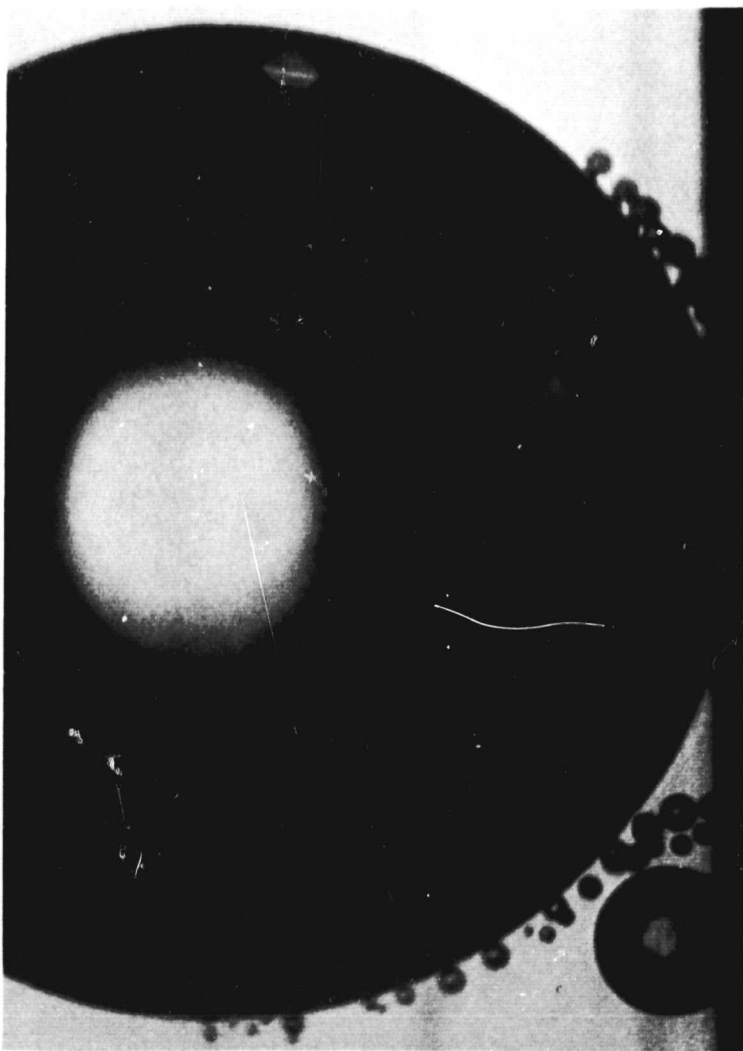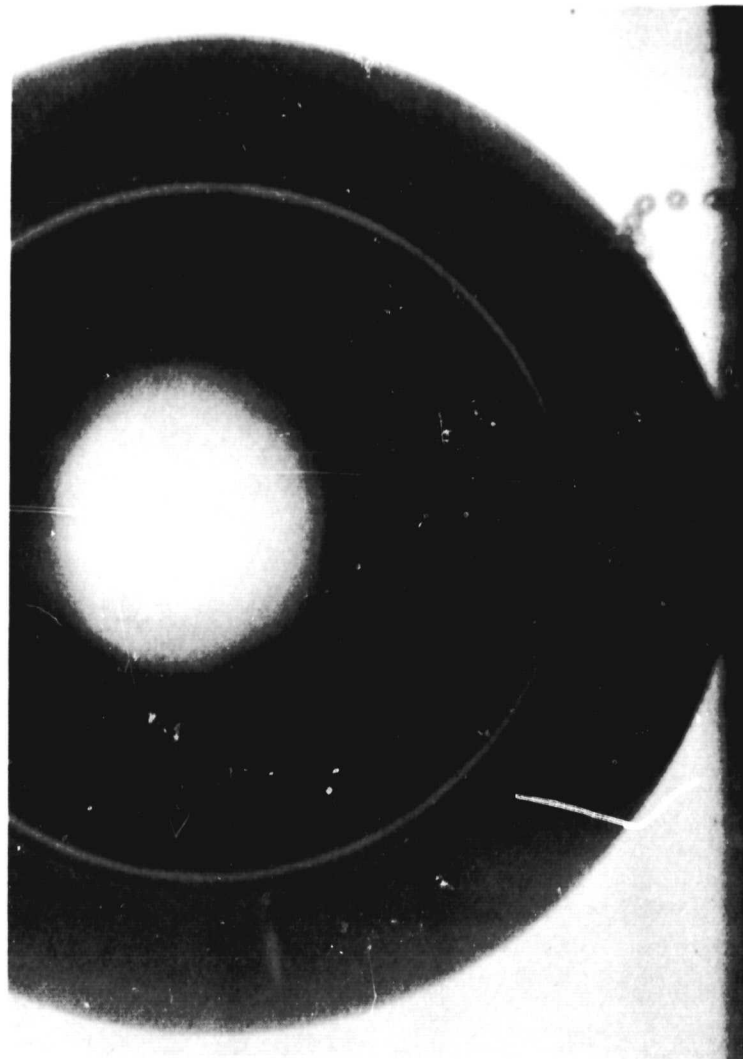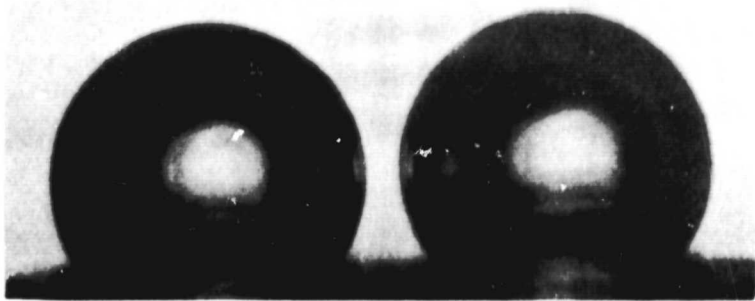   right= 19°

    Voltage= 1.58 V

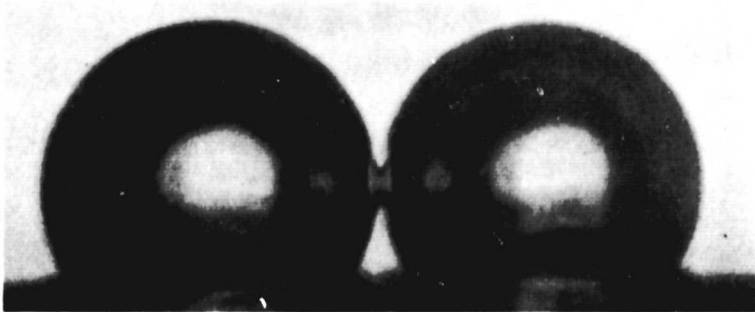Figure 9. Examples of bubble interaction in 1.0 N KOH solution.

depart due to gravity effects. Once the large bubble had
departed, the same sequence was repeated and a string of small
bubbles formed until finally one again began to grow to a
larger size. There were cases in which the second site was
farther away and kept producing small bubbles which de-
parted at the small size (0.0005 to 0.001 inch). These
bubbles did not coalesce with the larger bubble near its base
but rode up the curved surface, deflating all the time as the
larger bubble was fed in a manner similar to a balloon deflat-
ing, as can be seen in Figure 9(b).

During the runs with very low voltages, if there were
two equal sites just about a diameter away, the bubbles did
not always depart on contact and as can be seen from the se-
quence of photographs in Figure 10(a), (b), (c), and (d)
one bubble swung above the other, retaining contact but not
visibly feeding the other. Both bubbles then departed,
possibly because of the departure of the bubble still in con-
tact with the electrode. This phenomenon may be due partly
to the fact that the pressures inside the two similar bubbles
were nearly equal so that one bubble could not transfer gas
to the other due to the lack of a pressure difference. The
point of contact between the two bubbles is of particular in-
terest because it shows the nature of the neck joining the two
bubbles and may have a bearing on the electrode-bubble contact
characteristics.

While the experimental apparatus was being set up for
horizontal viewing of the electrode surface, photographs of
bubbles on a vertical cathode surface with a magnification of
500 times were obtained. These have been included as Figure 11

(a) Bubble diameter= 0.007 in. each
    Voltage= 1.81 V

(b) Bubble diameter= 0.0075 in. each
    Voltage= 1.81 V

(c) Bubble diameter= 0.008 in. each
    Voltage= 1.81 V

(d) Bubble diameter=
    0.008 in. each
    Voltage= 1.81 V

Figure 10. Bubble interference between nearly equal hydrogen bubbles
            in 1.0 N KOH solution.

(a) Bubble diameter= 0.003 in.   (b) Bubble diameter= 0.0035 in.
    Contact angle: left= 26°   Contact angle: left= 24°
                     right= 24°   right= 30°
    Voltage= 1.51 V   Voltage= 1.51 V

Figure 11.   Hydrogen bubbles growing 4.28 N KOH solution on a vertical surface.

(a) and (b) in this report because they confirm the work of Westwater, et. al., [24] in terms of the smallness of the contact angle and the bubble geometry.

IV. CONCLUSIONS

There are two distinct types of bubbles formed during electrolytic gas evolution. Bubbles having a departure diameter above 0.007 inch appear to have their maximum size controlled mainly by buoyancy forces while those in the range 0.0005 to 0.005 inch appear to be controlled by inertia forces.

The proportion of smaller bubbles to larger bubbles increases with an increase in current density although both types of bubbles form at all times.

The contact angle of the bubble is small, between 15° and 35°, and decreases as the diameter increases. The bubbles are nearly spherical throughout their growth on the electrode. Nowhere have they been found to be hemispherical, as has been assumed in some electrolytic bubble growth models, e.g., reference [14]. This may lead to significant variations between theory and experimental data.

The effects of bubble interference and interaction, especially at higher current densities are largely unknown and need to be studied and incorporated in the electrolytic gas bubble growth theories so as to give more realistic results. All the theories consider a single bubble surrounded by fluid, whereas in actual practice the neighboring bubbles can significantly alter the growth rate and the departure diameter.

V. FUTURE WORK

The investigations are to be continued to find the statistical distribution of the bubble size for other concentrations of acid and base electrolytes, for both hydrogen and oxygen. The contact angle and detachment diameter data for other electrolytes is also to be found and the variations from the present data studied.

In order to get a better understanding of the mechanism of nucleation, growth and collapse of the bubbles, a need for a continuous study of these processes has been felt. The use of a high speed movie camera would serve this purpose and the facilities for this that exist in the Boiling and Phase Change Laboratory are presently being used to set up the experimental arrangement. The Hycam high speed camera [Model 2004E] to be used can expose up to eleven thousand frames per second and it is expected that this would allow the study of the fastest growing bubbles as well as those that grow more slowly. The problems involved in the optics are presently being tackled and it is expected that full investigations will start shortly. Attempts are also being made to use higher resolution lenses so that finer details of the bubble base and contact area may be observed. This would enable more precise contact angle measurements to be made.

A start has already been made with the cooperation of Professor John H. Lienhard, who is studying the similarity of bubble growth in boiling and electrolysis under the Gravity

Boiling Project, NASA Grant NGR/18-001-035. The initial results
obtained from the high speed moving picture films are currently
being evaluated and will be presented at a later date.

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