S01.11) SURFACE WETTING AND THE DEPLOYMENT OF DROPS IN MICROGRAVITY

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ABSTRACT

The complete or partial deployment of liquid samples in low gravity is primarily influenced by the interracial properties of the specific liquid and solid materials used because the overwhelming bias of the Earth gravitational acceleration is removed. This study addresses the engineeri ng aspects of injecting and deploying drops of prescribed volume into an acoustic positioning chamber in microgravity. The specific problems of interest are the design, testing, and implementation of injector tips to be used in a simultaneously retracting dual-injector system in the Drop Physics Module microgravity experiment facility. Prior to release, the liquid to be deployed must be retained within a restricted area at the very end of the injectors under dynamic stimuli from the continuous injection flow as well as from the stepped motion of the injectors. The final released drop must have a well determined volume and negligible residual linear or angular momentum. The outcome of Earth-based short-duration low gravity experiments had been the selection of two types of injector tips which were flown as back-up parts. They were successfully utilized during the USML- 1 Spacelab mission as the primary tips. The combination of a larger contact surface, liquid pinning with a sharp edge, and selective coating of strategic tip surfaces with a non-wetting compound has allowed a significant increase in the success rate of deployment of simple and compound drops of aqueous solutions of glycerol and silicone oil. The diameter of the samples studied in the Drop Physics Module ranged between 0.3 and 2.7 cm. The tests conducted on-orbit with a manually operated small device have allowed the calibration of the volume deployed for a few drop sizes. The design for improved tips to be used during the next USML flight is based on these results.

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The shape of the free surface of liquids in contact with solids remains a subject of great interest especially when the overriding influence of gravity is removed [1,2] . In addition to a basic theoretical interest, the correct implementation of fluid management methods in low gravity relies, on our understanding of the interfacial statics and dynamics between liquids and solid surfaces [3]. Even when one is interested in the observation of the behavior of completely free three dimensional liquid surfaces, the. actual process of going from a contained liquid into a containerless environment is of primary importance. The study to be described below is a limited experimental investigation designed to develop, test, and implement a specific component for the controlled deployment of liquid samples in low gravity. It was designed as a low cost flight investigation in the framework of the USML- 1 Glovebox facility and in support to the on-orbit operations of the Drop Physics Module, an experiment facility dedicated to the study of the dynamics and of the surface rheology of free drops in microgravity.

1. THE PROBLEM

The objectives of the investigation of the behavior of free drops in low gravity can only be rigorously satisfied when these drops are actually deployed in a well controlled manner with well dc.fined volume and an uncontaminated surface. The weak acoustic positioning forces used in, the Drop Physics Module (l) PM) [4] will be effective at restraining the drop motion in a short amount of time only when the residual momentum imparted to the sample during the deployment process is negligible. The approach taken in this facility utilizes a pair of oppositely situated symmetrical injectors which simultaneously retract at high speed (> 10 cm/s) to leave a motionless drop in the center of the experiment chamber. This technique has been shown to be very effective when the liquid and solid deployment surface are characterized by a very large contact angle. The standard tips of the injectors are made of stainless steel and are 0.75 mm in diameter. 'l'he approach is thus to minimize contact between the drop and the injector tips by reducing their diameter to a minimum. "l'he drop volume of interest ranged between 0.S to 20 cm^3, *Figure 1a* is a picture of the opposed injectors with the standard tips as they are positioned in the center of the DPM chamber without a drop, and *figure lb* shows them holding a drop in the desired configuration in microgravity.

Even for reasonably high contact angle (about 90^{o} for distilled water and stainless steel) symmetrical wetting of both injector tips must be maintained in order to avoid spreading of the liquid over a single injector surface as shown in *figure* 2. In this case contact was broken with the upper left injector and the liquid spread over the lower right injector surface. The subsequent remerging of the two drops produced a liquid bridge anchored at the edge of the V-shaped injector housing.

The majority of the liquids used for the drop physics experiments during the USML- 1 Spacelab flight, however, displayed low contact angles with clean stainless steel surfaces. This made it quite difficult to maintain the configuration shown in figure 1 b under the continuing mechanical impulses due to the liquid injection and the stepped motion of the retracting injectors. liquids such as silicone oil and aqueous mixtures of inorganic surfactants (Sodium Dodecyl Sulfate) will spread over the contacting solid surface upon very slight impulses. *Figure 3 shows* a case where a glycerol-water mixture has spread over the injector tip and past the V-shaped edge in the case of the lower right injector. The subsequent deployment was asymmetrical as evidenced by the non-simultaneous breaking of the liquid necks (*figure 3c*). This asymmetry imparts linear momentum to the drop, and it results in translational oscillations of the sample in the acoustic pressure well,

The behavior of liquid bridges in low gravity has previously been investigated by others both during parabolic flights as well as during Spacelab missions [5,6]. Similar] y, the characteristics of liquid-solid interfaces relating to the aspects of liquid management have also been addressed in both static [7] as well as dynamic cases [8]. In this instance we are concerned with the additionat impact of the deployment dynamics on the behavior of the released drop. An additional constraint is imposed by the need for manual installation of **these** tips on the injectors inside the Drop Physics Module: because tips and injectors must be replaced after each experiment run because of cross-contamination concerns, very sharp edges must be avoided for crew safety reasons.

11, OBJECTIVES

The objectives of this flight demonstration were simple and limited in scope: The first task was to experimentally study the dynamics of drop injection, containment, and deployment in order to design specific injector tips which would minimize all detrimental impact. The second task was to manufacture a set of tips for use in microgravity with a manually operated apparatus to be used in the Glovebox facility in order to calibrate the deployed volume. An important aspect of this investigation was the ability to also use these tips in the DPM facility, and consequently, a third objective was to utilize these tips in case difficulty was encountered during deployment in microgravity.

111. GROUND-BASED DEVELOPMENT

The process of injection, stabilization, and deployment of liquid samples can be carried out in a 15 to 20 seconds time period for liquid volumes less than 5 cm^3. This has made it possible to perform experimental tests during parabolic flights of the NASA KC-13S airplane prior to the USML- 1 mission. The same manually operated mechanical drop deployment device built for the Shuttle Glovebox experiment was flown aboard the KC-135 to study the dynamics of drop injection and deployment with the standard small diameter injector tips as well as with a variety of custom-designed tips. *Figure 4a is* a photograph of the apparatus as operated in the KC-135, and *figure 4b* shows the same device on the Spacelab Workbench as it was configured for the experimental runs during the USML-1 flight.

Only distilled water and silicone oil (100 cSt viscosity) were deployed during the parabolic flight tests as they were considered the best and worst cases as far as drop deployment was concerned. All the tips tested were coated with a commercial surface treatment (trade name N yebar).

Distilled water did not spread over the coated small diameter (0.75 and 1.5 mm OD.) cylindrically shaped stainless steel and brass tips, and could be held between the opposing injectors and deployed without difficulty. The cohesive forces of water were able to overcome the adhesive forces between the liquid and the coated solid surfaces. This allowed the controlled deployment of water drops of up to 2 cm^3 in volume in an environment where the background residual acceleration was on the order of 0.05 G with transients on the order to 0.1 G (G is the magnitude of the Earth-based gravitational acceleration). *Figure 5* illustrates the effectiveness of the surface coating by showing the wetting of coated stainless steel tips by distilled water. Initially asymmetric wetting of the tips can be changed to symmetric wetting and positioning of the drop at the tip ends, allowing symmetric deployment of the sample.

Silicone oil, on the other hand, readily spread over the injector tip surface, and could not be deployed in a controlled manner by using small diameter cylindrical tips. Wider faced injector tips were thus designed in order to contain silicone oil and other spreading liquids, They allowed the demonstration of the stable capture of oscillating liquid bridges, and they also successful] y deployed silicone oil drops as shown in *figure* 6. Low gravity tests aboard the KC- 135 have therefore established that the fast retraction of opposed i njectors allowed the deployment of silicone oil drops of up to 2 cm^3 in volume with negligible residual moment urn even with tips having a significant contact surface area (18 mm^2).

This led to the selection of two tip designs for implementation in microgravity: type a tips have a flared out face of 9.5 mm in outer diameter and 1.75 mm in inner diameter and type **b** tips are cylindrical in shape with an outer diameter of 4.75 mm with a inner diameter of 1.55 mm. These two different designs are shown in *figure* 7 where they have. been mounted on the Drop Physics Module injectors. Both these types of tips have 9(I degrees sharply machined edges, and the flight units have been completely surface treated with a Nyebar solution through a dipping and bakeout procedure.

Figure 8 shows a series of sketches of other designs that we considered, but they were not chosen for various technical or safety reasons.

IV, EXPERIMENTS DURING THE USML-1 FLIGHT

Drop deployment experiments using the designed tips described above have been carried out both in the Drop Physics Module facility as well as on the Spacelab Workbench, Contrarily to initial plans, the tips were successfully used in the Drop Physics Module prior to tests in the Glovebox facility. Since feasibility had been demonstrated, the goals of the Glovebox demonstration were restricted to manual deployment and calibration of large drop volumes from 5 to 12 cm^3.

A. Spacelab Workbench tests

Initially scheduled at the beginning of the mission for the Glovebox facility, the drop deployment tests were postponed until the seventh day. They were carried out on the Spacelab workbench. The experimental set-up is shown in figure 4b, and the Orbiter video camcorder was used to record the experiments. The apparatus was gray-taped to the workbench surface, and existing Spacelab and workbench lighting was utilized.

The cylindrical liquid bridges had aspect ratio only up to 2.6, well below the Rayleigh limit. Very symmetric manual injection and deployment of water, of aqueous mixtures of glycerin, and of surfactant solutions was obtained. These deployments wereeithervideo recorded or transmitted live to the ground. *Figure 9* shows the sequence of injection and deployment of a 5 cm^3 water drop.

Detailed observation of the dynamics of drop deployment reveals that for larger droplet volumes (greater than 5 cm^3) the majority of the liquid remains undisturbed and motionless as the injectors withdrew symmetrically at high speed, 'I"he necks on both sides of the central drops elongate and break leaving no or several satellite droplets depending on the liquid viscosity. The small fraction of the liquid remaining on the injector faces (5 to 10%) appears to primarily depend on the size of the contacting surfaces and on the retraction velocity.

The results of the measurement of the deployed drop volumes arc displayed in table 1. These data have been gathered for distilled water and for the wide-faced tips. Three different desired volumes were investigated, and three different runs were carried out for each volume. The drops could not be captured in mid-air as planned, but they were retrieved from the walls of the Lucite chamber on which the liquid did not spread, thus allowing recovery of the entire sample. The volume measurements were obtained through a calibrated micrometer-driven syringe with an accuracy of 0.005 cm^3. The data obtained through this limited set of measurements indicate a volume reproducibility of +/-2 percent at worst. Part of this variation might be attributable to the fluctuation in retraction velocity inherent of the manually operated device. The stepper motor-controlled injection system of the Drop Physics Module provides more reproducibility in the retraction velocity as well as higher rates.

B. Drop Deployments in the Drop Physics Module

Both types of tips were successfully used during the experiment runs of the DPM facility for the deployment of simple as well as compound drops (a drop within a drop) of aqueous mixtures of glycerol, silicone oil, and hydrocarbon compounds. The abrupt 90° edges of the tips contained the liquid within the required surfaces during the first injection and deployment, but the sudden stop at the end of the injector retraction invariably drove the liquid beyond the pinning edge, and allowed the spreading of the liquid over the body of the tips. The liquid containment for silicone oil was still so precarious that in one particular instance the bursting of a bubble caused the liquid to spread beyond the pinning edge.

Figure 10 displays a series of video frame recording of a water-gl ycerol mixture of 50 cSt viscosity deployment. The cylindrically shaped tips (type **b**) were used. Near perfect symmetry can be observed by noting the breaking points of the necks on each side of the drop as well as the symmetrical shapes of the residual shape oscillations, ***Figure 11*** is a similar series, of video frames depicting the deployment of a 100 cSt silicone oil. The wide-faced injector tips (type **a)** were used. Good symmetry can be observed once again, The deployed sample remained motionless in both instances,

Type **b** tips were also successfully used to inject and to deploy compound drops of a water-glycerol solution into a silicone oil drop.

V. CONCLUDING REMARKS

liquid wetting and spreading acquire greater importance in microgravity when one becomes concerned with containment and material contamination management. Notoriously difficult to handle liquids such as silicone oil and surfactant solutions must be restricted to limited areas by physical barriers such as a sharp pinning edge. Although not foolproof, this method appears to be effective in normal acceleration conditions of the Space Shuttle. In the restricted area of drop deployment, the results of these low gravity tests show that a compromise involving a larger liquid-solid contacting area will still allow the reproducible deployment of drops with controlled volumes. Because orbital operation time is extremely valuable, drop deployment procedures that minimize liquid clean-up and sample translational oscillation damping times must be adopted.

improvements based on these results will involve the elimination of sudden acceleration pulses in the direction tending to induce liquid spreading, sharper pinning edges (30 or 45° instead of 90°) with a safe installation procedure, slower liquid injection rates or larger injector bores, and finally real-time crew interaction with the injection/deploynlcnt mechanism.

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FIGURE CAPTIONS

Figure 1:

1a. Small diameter standard DPM injector tips and housing as viewed along the Z axis of the experiment chamber

1 b. Nominal configuration for a drop to be deployed, "l'he small contact surface between the tips and the drop and the symmetrical retraction of the injectors insure that a very small fraction of the liquid remains on the injector tips and that minimal momentum is imparted to the released sample.

Figure 2:

Still video frame sequence showing the loss of contact with one injector tip and the subsequent spreading of the liquid (distilled water) all the way to the injector housing collar (V-shaped solid surface). For a non-spreading liquid like water, this can be prevented by maintaining a larger contact surface with both tips.

Figure 3:

in these video frames a water-glycerol mixture has spread past the V-shaped housing to the O-ring on one side of the lower-right injector (a). Subsequent deployment creates an initial time lag in the breaking of the liquid neck connecting the drop to the more wetted injector (b and c).

Figure 4:

4a. Manually operated Glovebox demonstration apparatus as installed on a breadboard during short duration low gravity tests aboard the NASA KC- 135 aircraft.

4b. 'I'he same apparatus installed on the Spacelab Workbench during the USML- 1 mission. The apparatus was taped to the work surface, and the operations were recorded and downlinked to the POCC using the Orbiter 8 mm video Camcorder.

Figure 5:

Video frame sequence showing the effectiveness of treating the solid surface with a non-wetting coating. From an initially asymmetrical attachment, the solid-liquid interfaces have been moved by the retracting injectors to allow a near-symmetrical positioning of the liquid bet ween the injector tips.

Figure 6:

6a, Successful anchoring of silicone oil by a flared tip surface with 900 sharp edge. The liquid bridge is stable even under the acceleration perturbations characteristics of the KC-135 flights.

6b. Deployment of silicone oil drops has been demonstrated in low gravity aboard the KC-135 . The deployed sample is released at the center of the initial liquid bridge with minimal residual linear momentum.

Figure 7:

The two principal designs for the Glovebox demonstration tips as installed on the DPM injectors. Type **a is** a flared surface tip 9.5 mm in outer diameter and type **b** is cylindrically shaped with 4.75 mm in outer diameter. Both have been treated with a chemical non-wetting agent, and have a sharp 90 degrees pinning edge.

Figure 8

Sketches of other designs of injector tips tested during the development phase. 1. This design has the same outer diameter as the flown type b tips, but is made of thin-walled stainless steel tubing. It was not implemented because of the very small anchoring surface perpendicular to the tip body. 2. This design is the same as 1. except for a smaller outer diameter intermediate between 1. and the standard DPM tips shown in figure 1. 3. This design was found to perform as well as the type **a** tips selected. It was not selected due to safet y concern about the sharp 45 degrees edges. 4. This design was tested, and the central protruding tip was eliminated to obtain the flight type **a.**

Figure 9:

Video frame sequence showing the deployment of a 5 cm^3 water drop by the manually-operated apparatus on the Spacelab Workbench. Measurement of the deployed volume has been obtained by retrieving the sample after impact on the lucite walls, As shown by Table 1,5 to 10% of the injected volume remains on the tips, or is lost to the deployed drop through non-coalescing satellite droplets.

Figure 10:

Video frame sequence of the top view of the deployment of a 50 cSt water-glycerol mixture using the type **b** injector tips in the DPM. Ne.ar-perfect symmetry can be observed by the shape of the drop and the simultaneous breakage of the two necks joining the liquid sample to the injector tips.

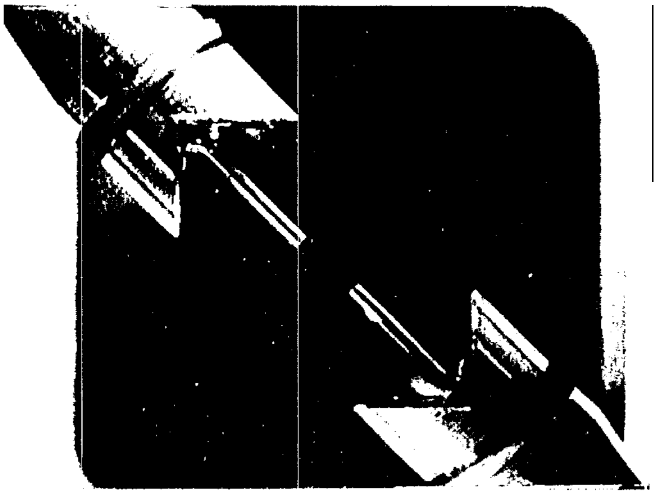
Figure 11:

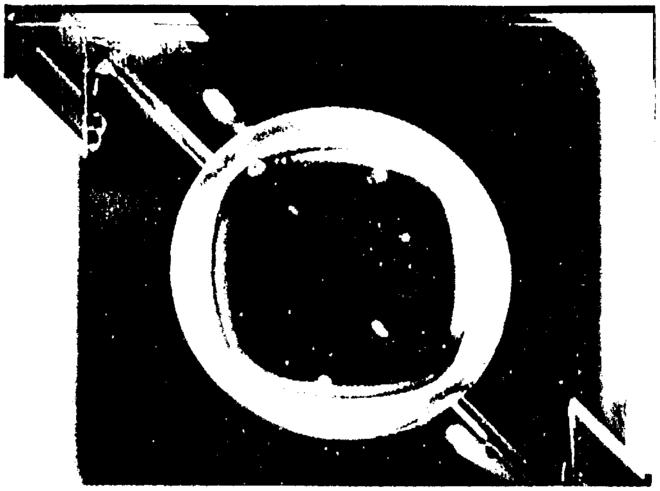
Video frame sequence showing the details of the deployment of a 100 cSt silicone oil drop using type a injector tips in the DPM.

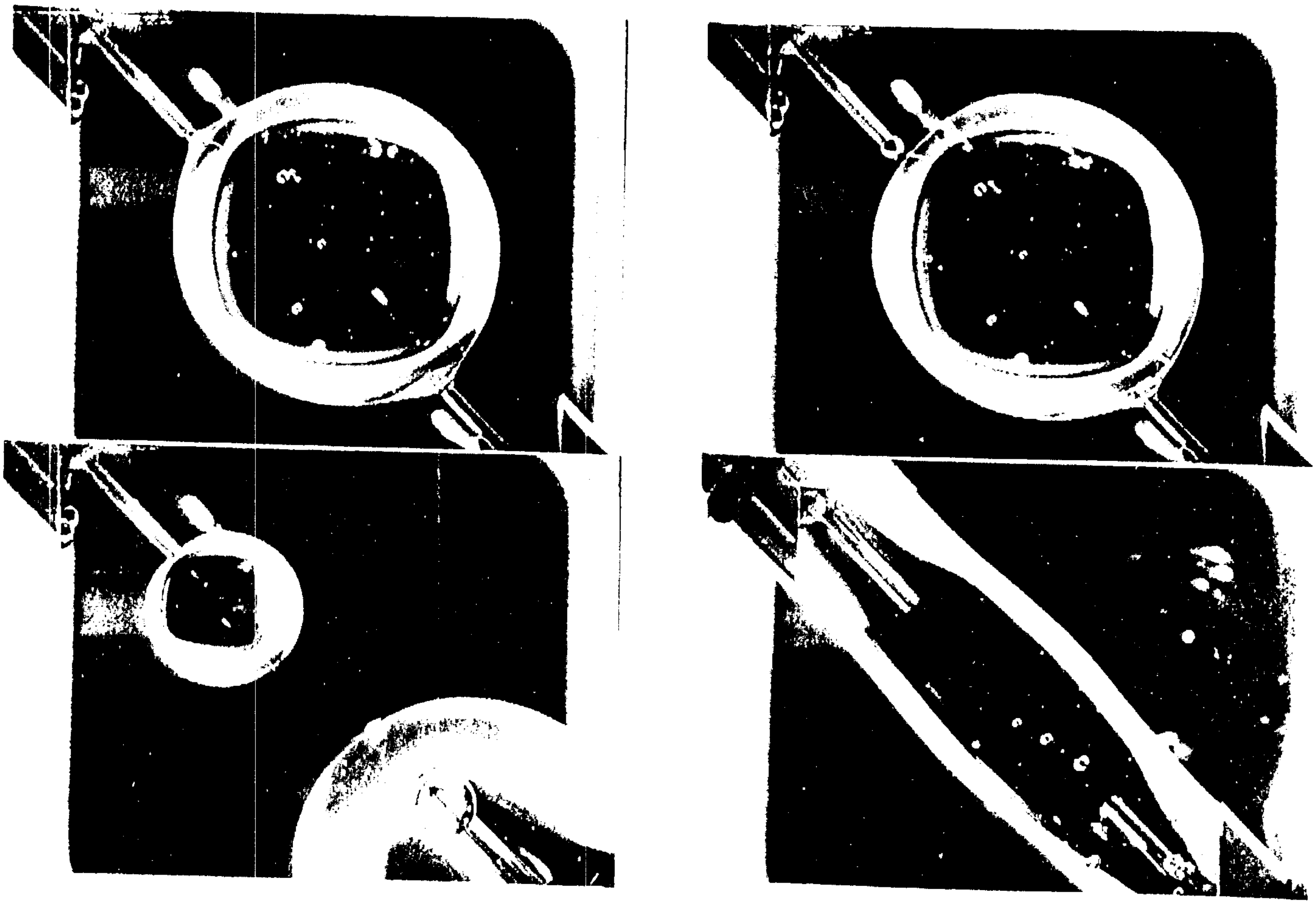
TABLE I

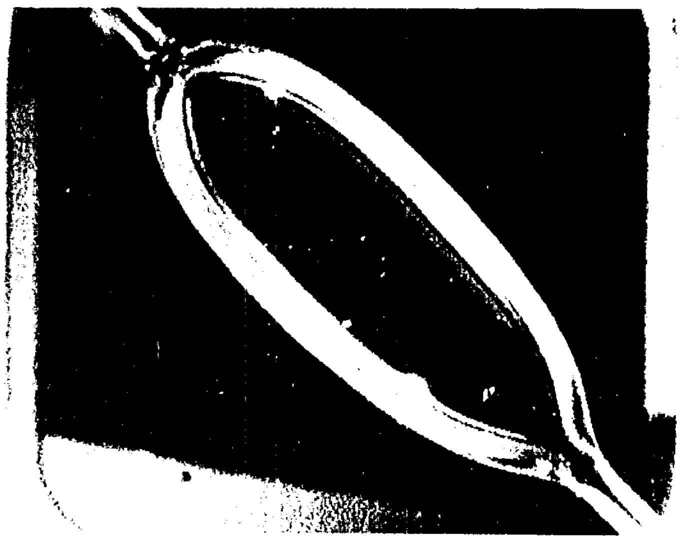
Results of the measurement of the deployed drop volumes. The samples were retrieved with a micrometer-driven syringe, and the measured volumes are listed together with the desired injected volumes.

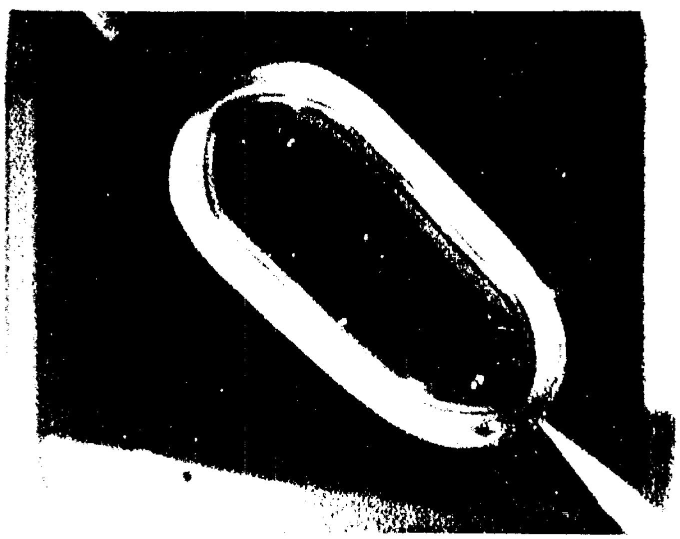
INJECTED VOLUME cm^3	DEPLOYED VOLUME cm^3
5	4.45
5	4.35
5	4.52
7	6.28
7	6.20
7	6.30
12	11.65
12.	11.45

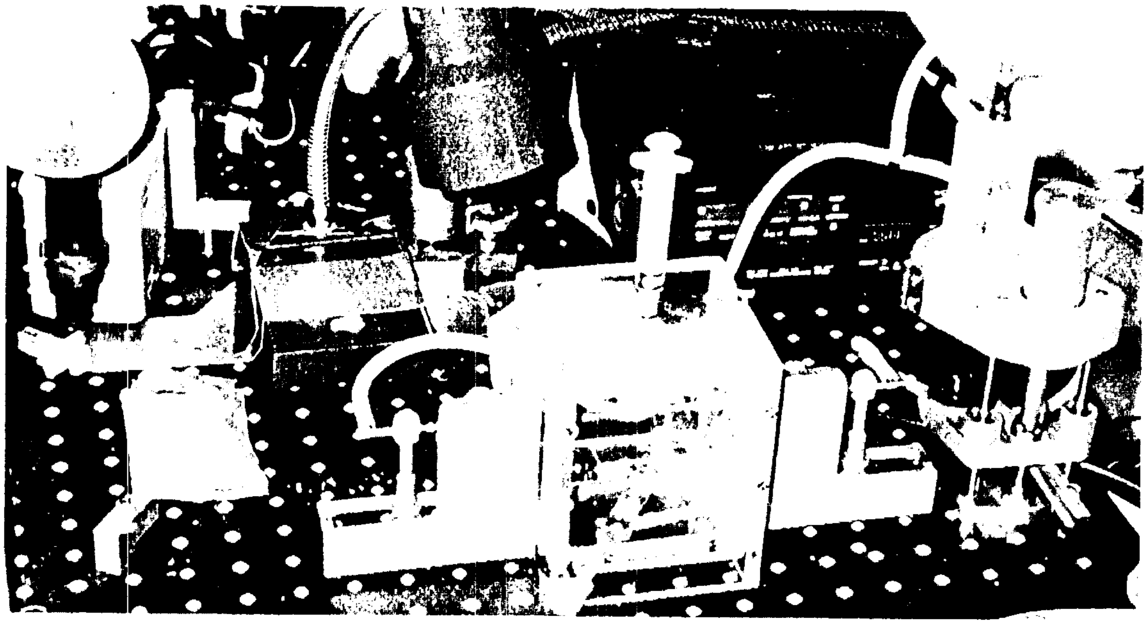


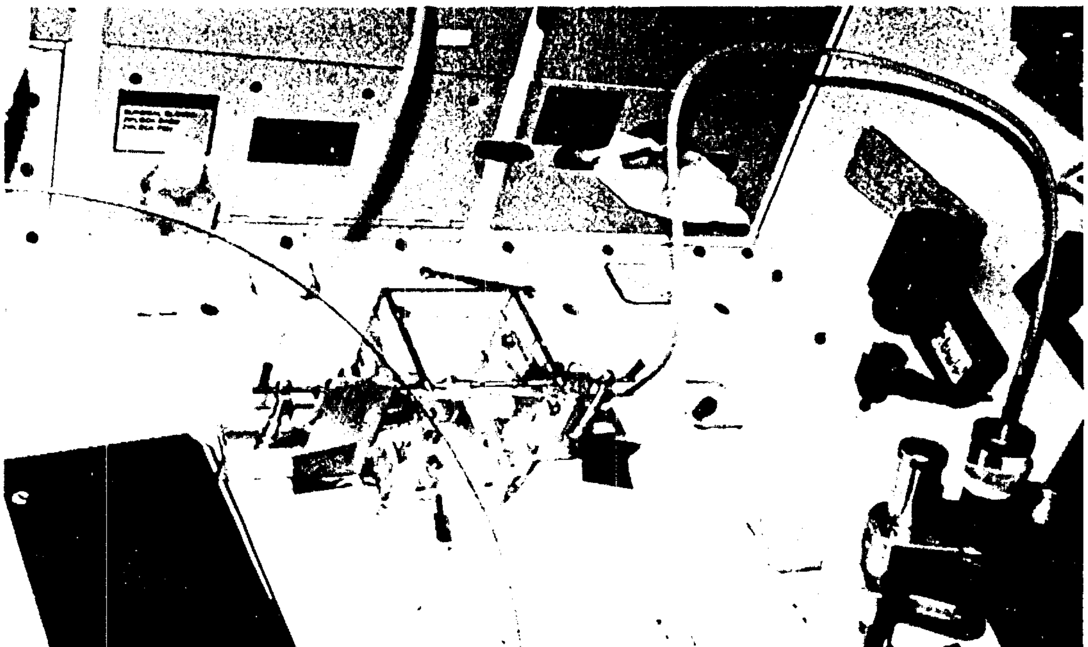


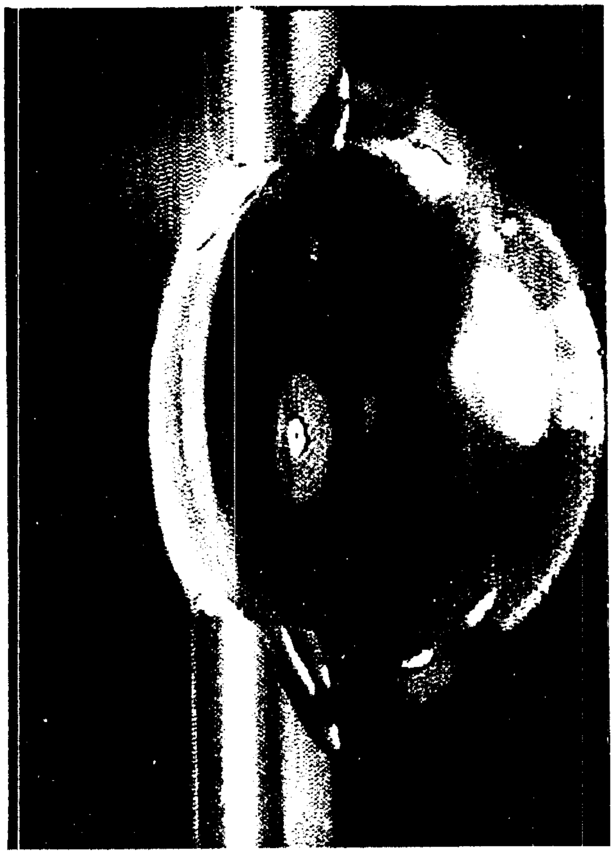
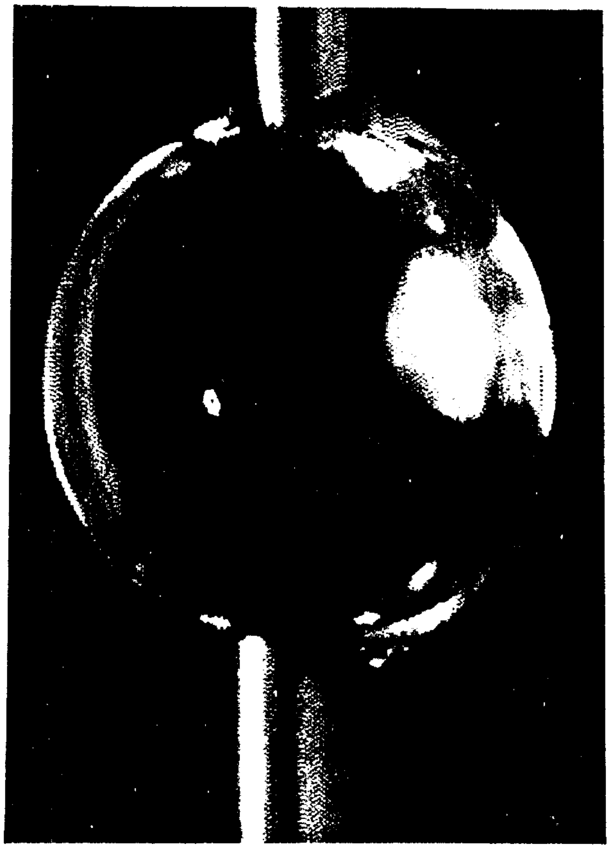
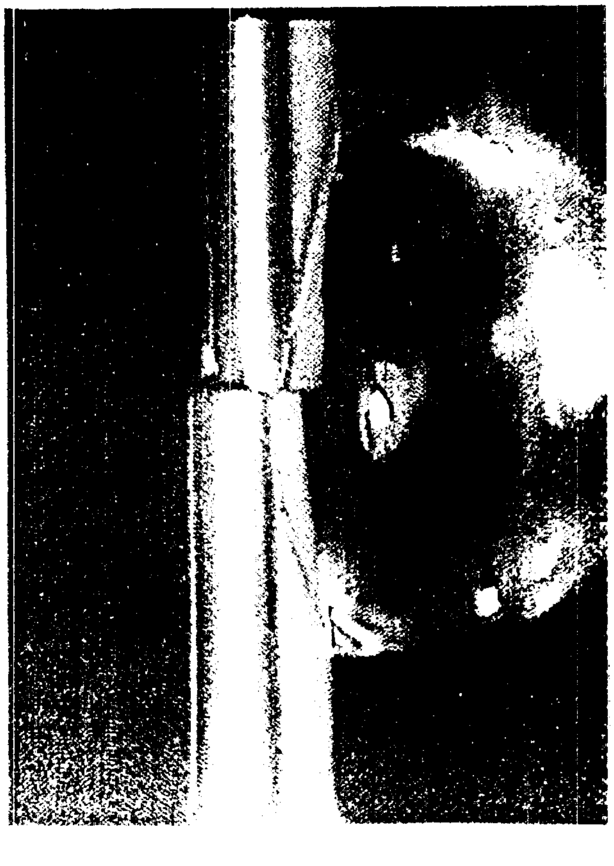
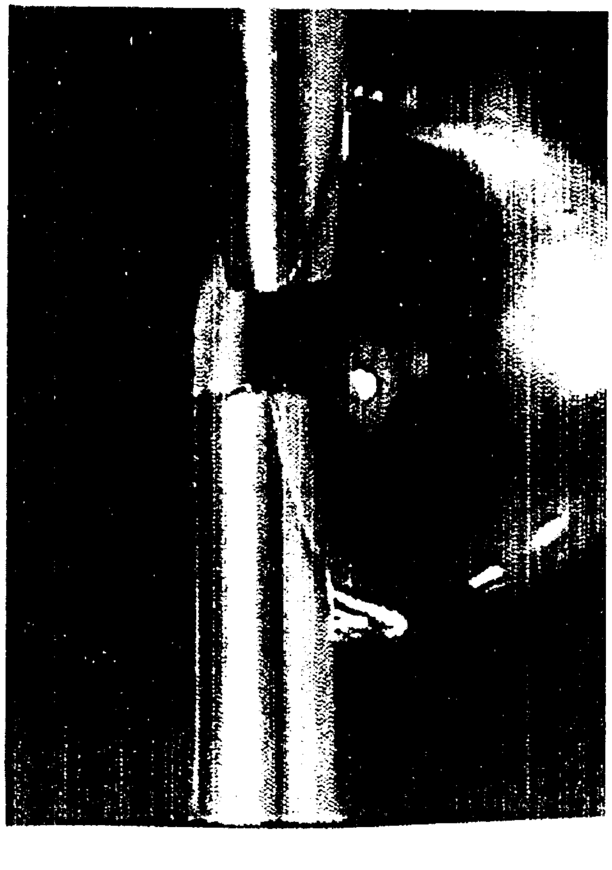


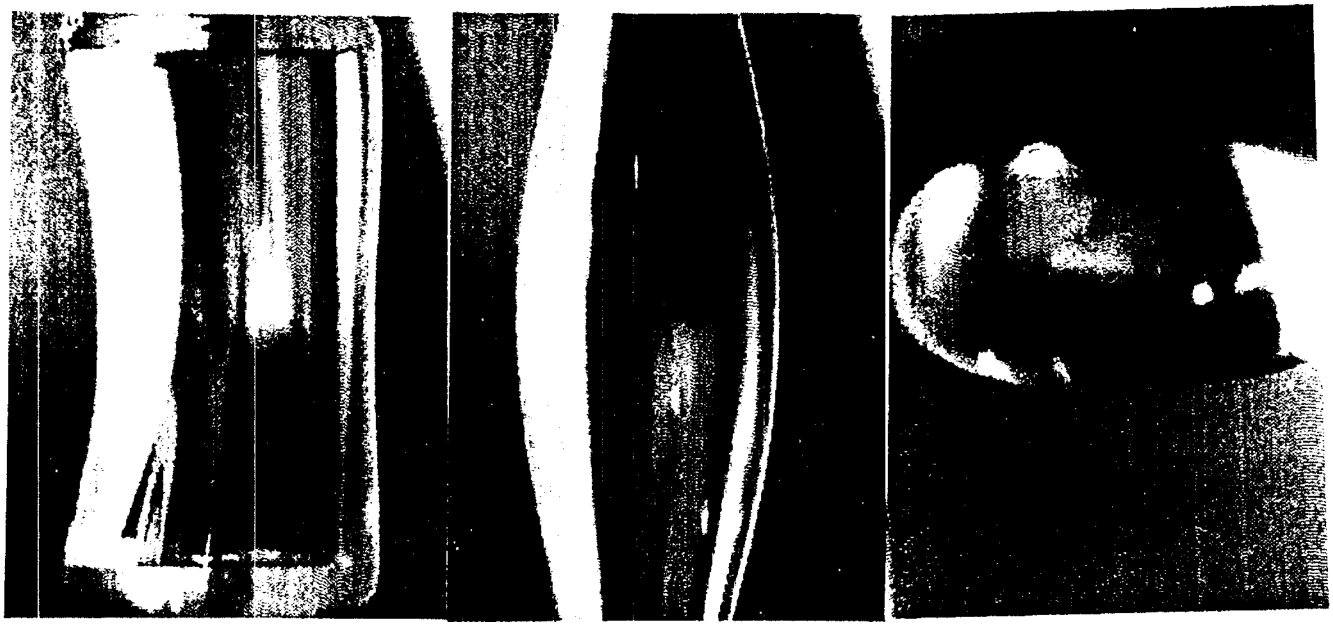


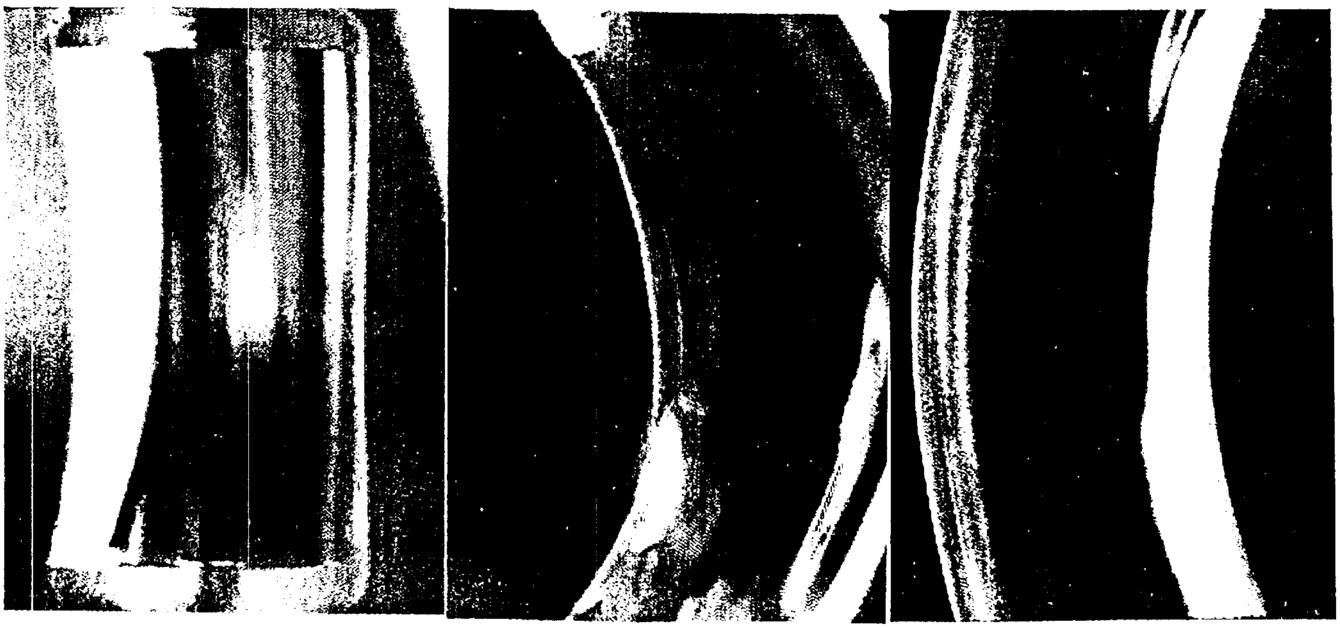


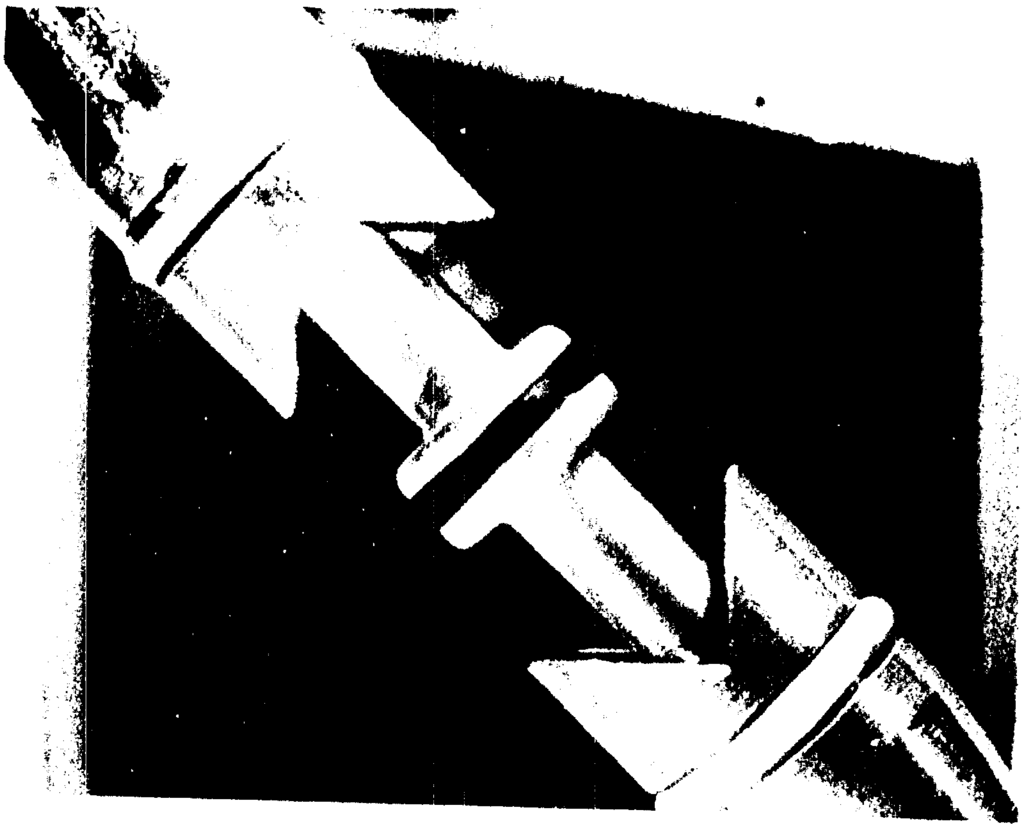


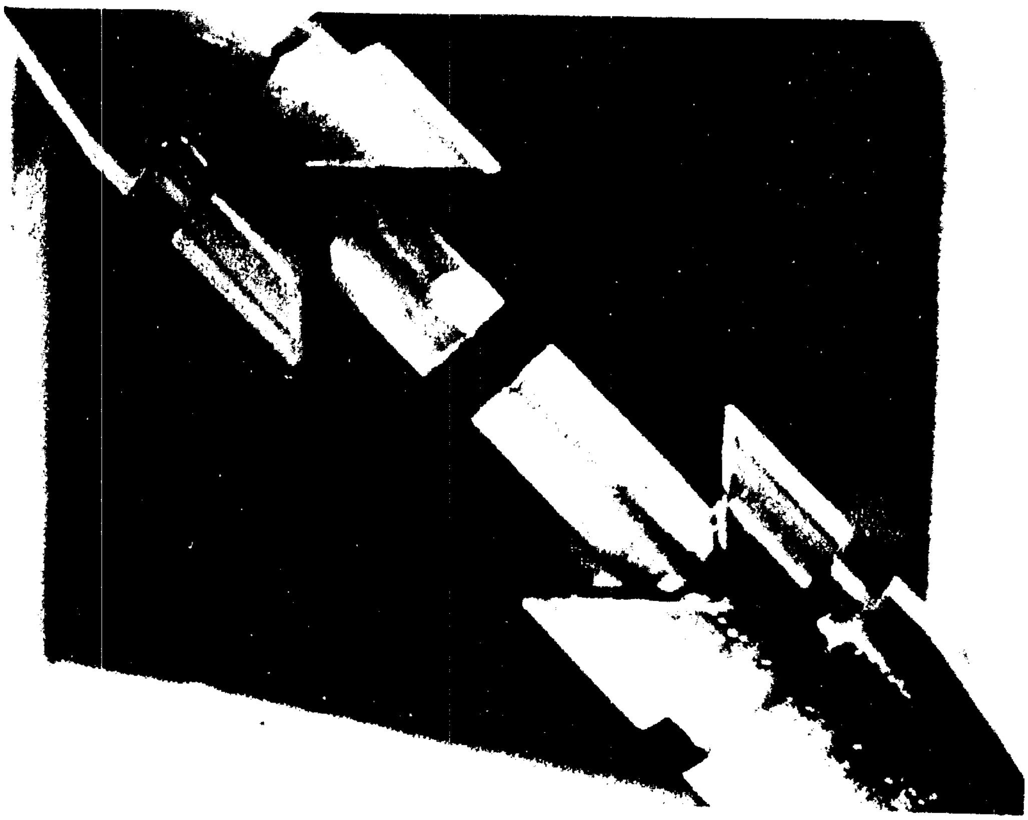


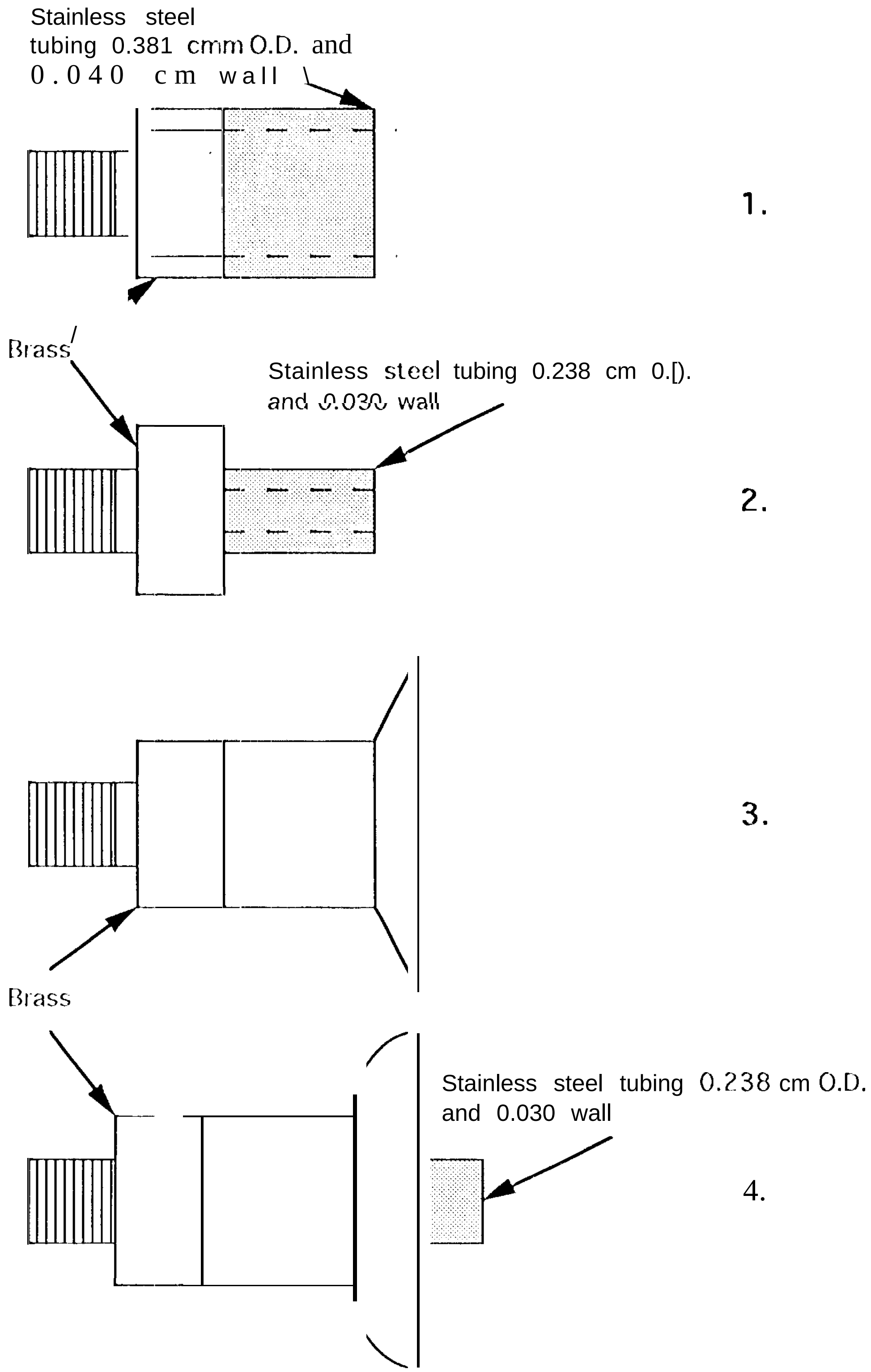














Stainless steel
tubing 0.381 cmm O.D. and
0.040 cm wall
1.
Brass
Stainless steel tubing 0.238 cm 0.D.
and 0.030 wall
2.
3.
Brass
Stainless steel tubing 0.238 cm O.D.
and 0.030 wall
4.