PHASE PARTITIONING, CRYSTAL GROWTH, ELECTRODEPOSITION AND COSMIC RAY EXPERIMENTS IN MICROGRAVITY

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ABSTRACT

Five experiments are contained in one GetAway Special Canister (5 ft^3). The first utilizes microgravity to separate biological cells and to study the mechanism of phase partitioning in 12 separate cuvettes. Two experiments are designed to grow organic crystals by physical vapor transport. One experiment consists of eight electroplating cells with various chemicals to produce surfaces electroplated in microgravity. Some of the surfaces have micron sized particles of hard materials co-deposited during electrodeposition. The fifth experiment intercepts cosmic ray particles and records their paths on photographic emulsions. The first four experiments are controlled by an on-board C-MOS controller. The fifth experiment is totally passive. These are the first of a series of experiments planned by the Consortium for Materials Development in Space. Their purpose is to create new commercial products with microgravity processing.

INTRODUCTION

The GAS Experiment package consists of five experiments. Four utilize the microgravity of space flight and one is a cosmic ray experiment. The experiments are: Separation of Aqueous Phases, J. M. Harris principal investigator (P.I.) cooperating with Celanese Research Corporation; Polymer Crystallization, S. P. McManus, P.I. cooperating with General Telephone and Electronics; Monomeric Crystallization, J. M. Harris, P.I. cooperating with Celanese Research Corporation; Electrodeposition and Co-deposition, H. D. Coble, C. Riley and Boon Loo co-P.I. cooperating with McDonnell Douglas Astronautics Company-Huntsville; and Cosmic Ray studies, J. C. Gregory, P.I. The Marshall Amateur Radio Club has prepared an electronics package to support the experiments thereby providing power, control, and a data system. Graduate and undergraduate students participate in the design and testing of the apparatus.

SEPARATION OF AQUEOUS PHASES (SOAP)

When aqueous solutions of pairs of certain neutral polymers (such as dextran and polyethylene glycol) are mixed above certain concentrations, systems composed of two immiscible phases are formed. If an impure biological material is added to such a system, and the system shaken, the biomaterial will, upon re-equilibration, partition between the two phases or, in the case of cells, between one of the phases and the interface between the phases. This simple phase partitioning process has been applied as a very important purification technique of considerable biotechnological importance.

Gravity plays a major role in phase partitioning. Not only does gravity degrade separations via the slow sedimentation of cells, but it also affects partition by controlling the rate of phase separation and the resulting fluid convection.

The SOAP equipment consists of a set of small cube shaped glass cuvettes (approx. 1.5 ml) with a magnetic stirring bar inside and a small stirring motor on top. These stirred cuvettes are monitored by means of 35 mm photography.

A set of twelve cuvettes are mounted in a temperature controlled, insulated aluminum block. See figure 1. The back of the block contains a frosted glass window through which a strobe light flashes for back lighting the cuvettes. The front of the block contains a clear piece of glass for an unobstructed view of the cuvettes. The glass has an infrared reflective coating to decrease radiation heat loss from the cuvettes. A nichrome wire spans the space between the cells and acts as a resistance heater. The temperature is sensed by the controller via a thermistor in the block; the controller regulates power to the nichrome wire. Insulated covers enclose the remainder of the block.

The object of this microgravity experiment is to study the rate of phase separation in low G and determine means by which the rate may be controlled. This study builds on a previous UAH shuttle flight (STS 51D) in which preliminary experiments were performed. It appears that wall coatings can be used to accelerate and control phase separation. This first flight tests this tentative conclusion. Coatings are applied to some of the glass cuvettes and stirring bars.

The actual performance of the experiment is simple. The device is designed so that upon activation by the G #105 on board controller, the two immiscible liquids are stirred for 20 seconds (as determined by testing). Then the stirring stops and photography begins. Photographs are taken every 30 seconds for four minutes followed by photographs every 10 minutes for two hours; a final photograph is taken at 10 hours. These photographs will later be analyzed with an optical densitometer.

POLYMER CRYSTALLIZATION

Crystals of a monomeric organic substance are grown by vapor phase deposition. The experimental compartment consists of a series of quartz reaction cylinders. See Figure 2. A fritted glass disk in the center of each of the cylinders divides the chambers and physically retains the uncrystallized mate-

rial in one end of the cell. The cylinders are under high vacuum and the fill tube is removed for flight. The material to be vapor deposited is coated on glass honeycomb in one end of the cell, which has a resistance heater wound around it. At the other end of the cylinder a seed crystal of the material is mounted. That end is fitted with a thermoelectric cooling device. Temperature monitors are fitted at the two ends of each tube. A thermocontroller portion of the on-board controller is utilized to control the temperatures.

When the experiment is turned "on" by the G #105 on-board controller, a signal is given the thermocontroller to control the cell temperature containing the monomeric material at about 80 degrees C. At the same time, the thermocontroller controls (by cooling) the end of the cell containing the seed crystal so that its temperature is 80 degrees C lower than that of the heated end of the tube. When both ends are at their target temperatures, vapor or organic material will pass from the heated cell to the cooled cell and crystallize on the seed crystal resulting in the growth of a large single crystal. After an appropriate growth period, presently estimated at 16 hours (time to be established in ground-based experiments by GTE) the thermoregulator will be instructed to cease cooling and heating the device.

The resulting crystals, to be collected, polymerized and analyzed after the GAS can is opened are of a type which have great promise for application in a new generation of high speed computers and in the communications industry.

MONOMER CRYSTALLIZATION

In this monomeric crystallization experiment (MCE) organic crystals are grown by vapor deposition. The organic materials are provided by Celanese Corporation and are of great interest as nonlinear optical materials. It is expected that the absence of concentration driven convection at the face of a growing crystal results in growth of more highly ordered and possibly larger crystals. Previous experiments have shown that improved crystal size and quality is achieved in microgravity. This experiment builds on previous experience to grow more highly ordered and larger organic crystals for optical applications. After crystals are recovered they will be evaluated by Celanese and by the Materials Sciences Laboratory at UAH.

The experimental compartment consists of an evacuated quartz tube, similar to that used in the polymer crystallization experiment. See Figure 3. Again, the fill tube is removed before flight. The entire tube is wrapped with resistance heating coils. The tube end containing the material to be vapor deposited is heated to within 3-5 degrees (T_1) of the melting point (T_m) while the deposition end (containing a seed crystal on a glass flat) is heated to T_2 (to within 2-5 degrees of T_1). Thus,

$$T_1 - T_2 = 2\text{-}5^\circ\ C$$

$$T_m - T_1 = 3\text{-}5^\circ\ C$$

Two materials are under consideration having T_m values of 133 and 90° C. These melting points define the temperature requirements of the apparatus. Temperatures are measured during the experiments by thermistors at both ends of the tube and in the middle. These temperatures are stored and controlled as a function of time during the experiment by the on-board computer.

The time required for the experiment is approximately 24 hours depending on the temperature difference and material chosen. When the experiment is turned on by the G #105 on board controller the tube containing the material to be vapor deposited is heated to T_1 while the other end of the tube is heated to T_2. Vapor of the material passes to the cooler end where it crystallizes on the seed crystal resulting in the growth of a large single crystal. After an appropriate growth period the experiment is turned off and the crystals returned to earth for evaluation.

ELECTRODEPOSITION AND CO-DEPOSITION

Electrodeposition and co-deposition of materials are commercially important processes which can produce superior products under low gravity conditions because electromechanical processes are significantly influenced by gravity. The objective of this experiment is to understand gravity's role in simple metal electrodeposition and co-deposition for the purpose of producing commercial products in space. Surface coating processes to be developed for application in industrial operations are: 1) coatings for producing crystal morphologies with catalytic properties significantly greater than that of metal catalysts produced on Earth; 2) coatings that maximize wear-resistance, antifriction and anti-seizing properties.

During electrodeposition in a gravity environment, density gradients are created which result in convective transport that affects forming and surface preparations. Similarly, in co-depositions, particles in neutral suspensions tend to sediment, causing inhomogeneous coatings and reductions in the volume fraction of the occluded neutral. The microgravity available in Earth orbit provides the environment needed to develop electromechanical processes producing more homogeneous commercial products.

Catalytic activity is related to the size of the catalyst particles and their particular morphologies. The crystallite morphologies in face-centered cubic metals appear to offer the best promise of increased catalytic activity for certain chemical reactions. The higher the deposition potential, the finer the crystalline deposit; thus, a product with high catalytic activity results. In low gravity, the suppression of gravity driven convection will allow the electrodeposition of more powdery, dendritic and twinned deposits, and hence, more active catalysts than are possible on the ground.

We are studying the electrodeposition of materials in space, using eight electrodeposition cells per flight. See Figure 4, and also refer in this volume to a companion paper entitled "Microgravity Effects on Electrodeposition of Metals and Metal Cement Mixtures" by Maybee, Riley, Coble. Metals which may have commercial applications as catalysts include: platinum, palladium, gold, silver, iron and cobalt. We are also testing the feasibility of electrodepositing a metal and a refractory (cobalt-Cr_3C_2)in the low gravity environment of

space. The cells (See Figure 5) contain solutions of soluble salts of the metal ions to be deposited. Some of the cells contain micron size particles of chromium carbide or diamond dust. Each cell is energized with a voltage source, each cell drawing approximately 20 milliamps or less of current for up to six hours. The cells, along with their milliammeters are photographed during electroplating. A digital thermometer displays the temperature next to the cells and one cell's voltage will be monitored. These too are photographed.

When the experiment is turned on by the G #105 on-board controller, the two upper co-depositing cells are stirred. After five minutes, the voltage is applied to the electrodes in the cells. Photographs are taken at selected time intervals for six hours. The controller regulates the temperature to a minimum of 25° C during the experiment and to a minimum of 5° C before and after the experiment to prevent freezing. The controller records the temperature, current and voltage and initiates the camera and flash photography. The electrodes are then analyzed and tested upon return to earth.

COSMIC RAY DETERMINATION

Photographic emulsions have been used for 50 years for cosmic ray and particle physics research. It is expected that large emulsion calorimeters will be flown on the Shuttle or Space Station to develop further these areas of high energy physics which UAH and Japanese American Cooperative Emulsion experiment collaboration have recently pioneered.

Fine tuning of the design of these multi-element passive detectors requires a realistic determination of the particle background induced in a massive calorimeter detector. A small emulsion calorimeter, approximately 15 cm by 15 cm is to be flown in the UAH GAS-can to provide this assessment. This experiment also determines materials suitable for marking nuclear tracks.

The emulsion chamber instrument of the type proposed here is totally passive, and consists of approximately 150 thin plates of material, each 6 inches x 6 inches, assembled in a stack 6 inches high. The stack of precision machined plates is mounted in a rigid, lightweight box. The material of the plates includes: silver bromide gel, polymethyl methacrylate (lucite), polycarbonate resin (CR-39), x-ray film, and lead or tungsten plates. These materials are hermetically sealed in the emulsion chamber box, which is placed in the GAS-can so that there is minimum shielding between it and space. The photographic emulsions will be analyzed for nuclear markings upon return to earth.

ACKNOWLEDGEMENT

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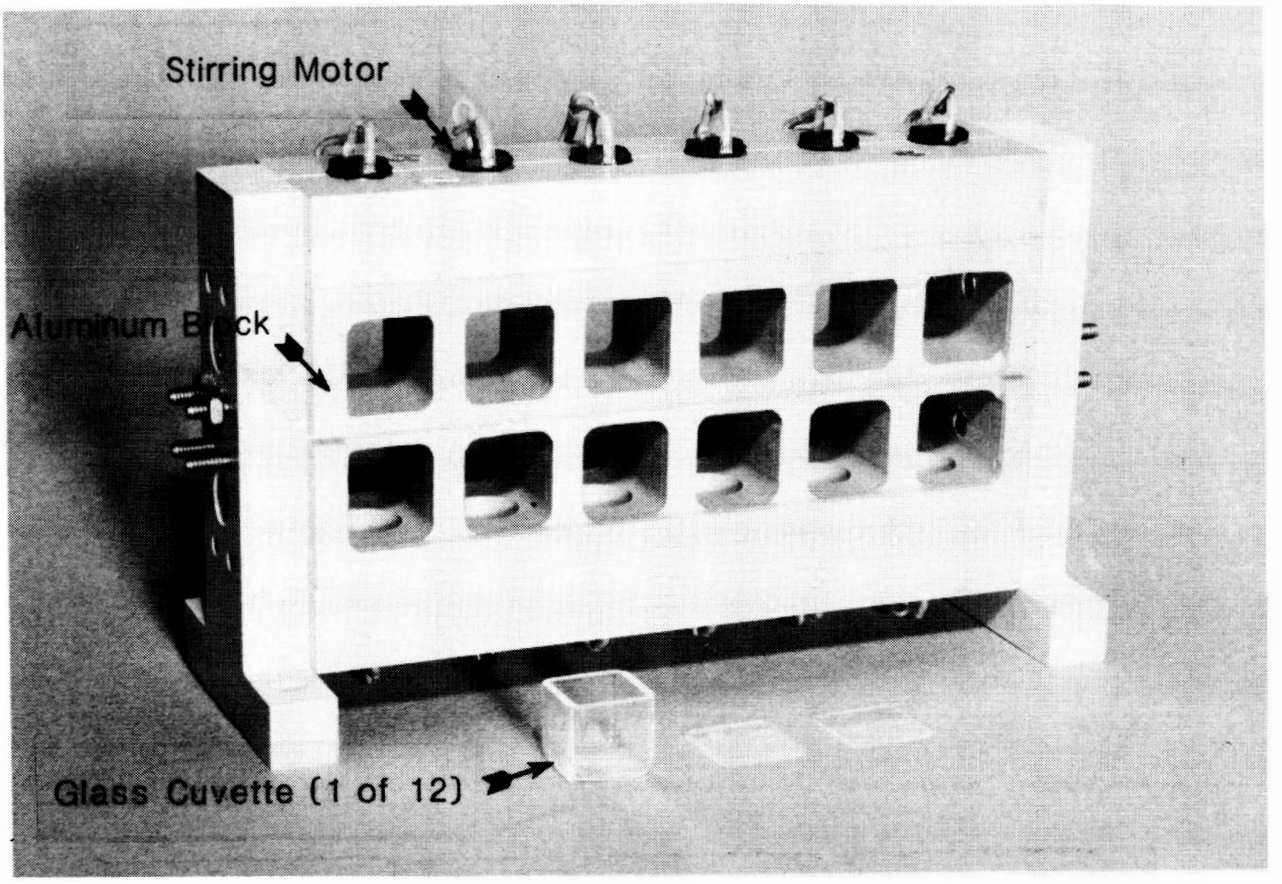

Figure 1 Separation of Aqueous Phases Apparatus

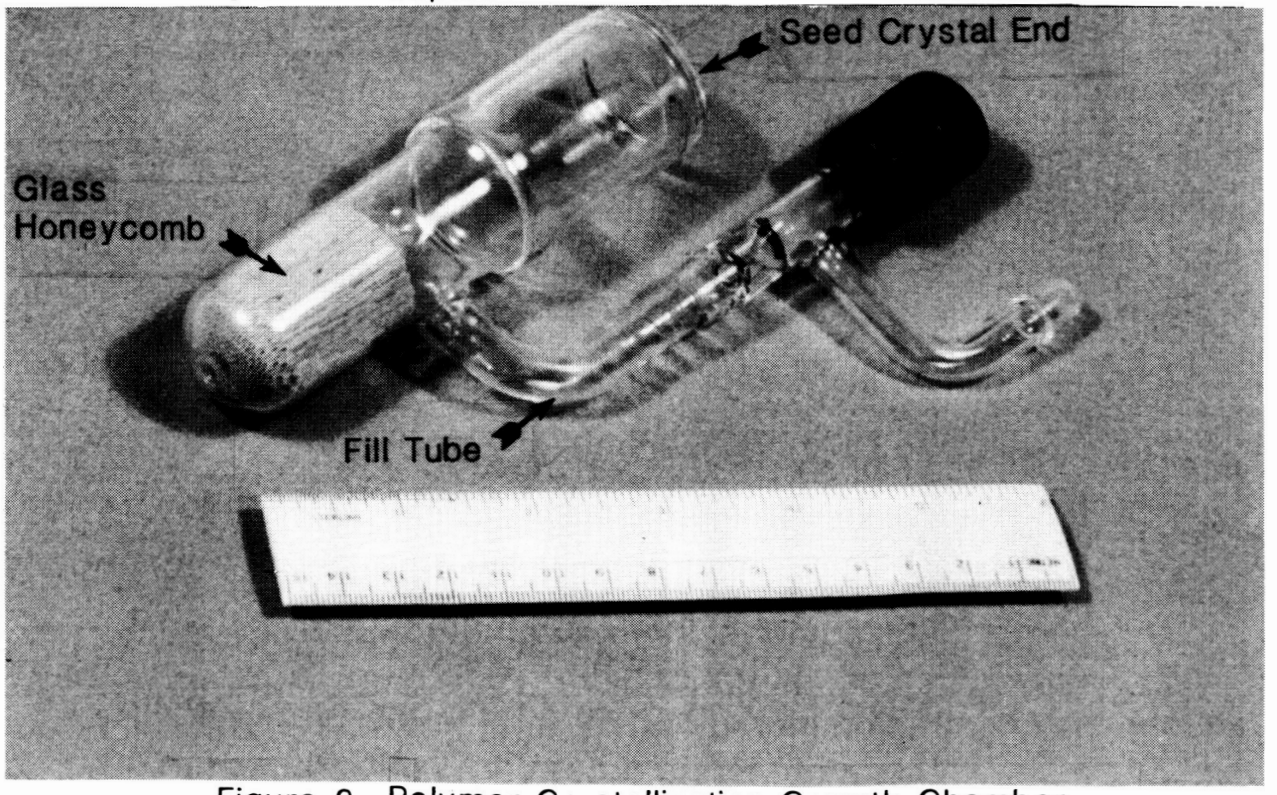

Figure 2 Polymer Crystallization Growth Chamber

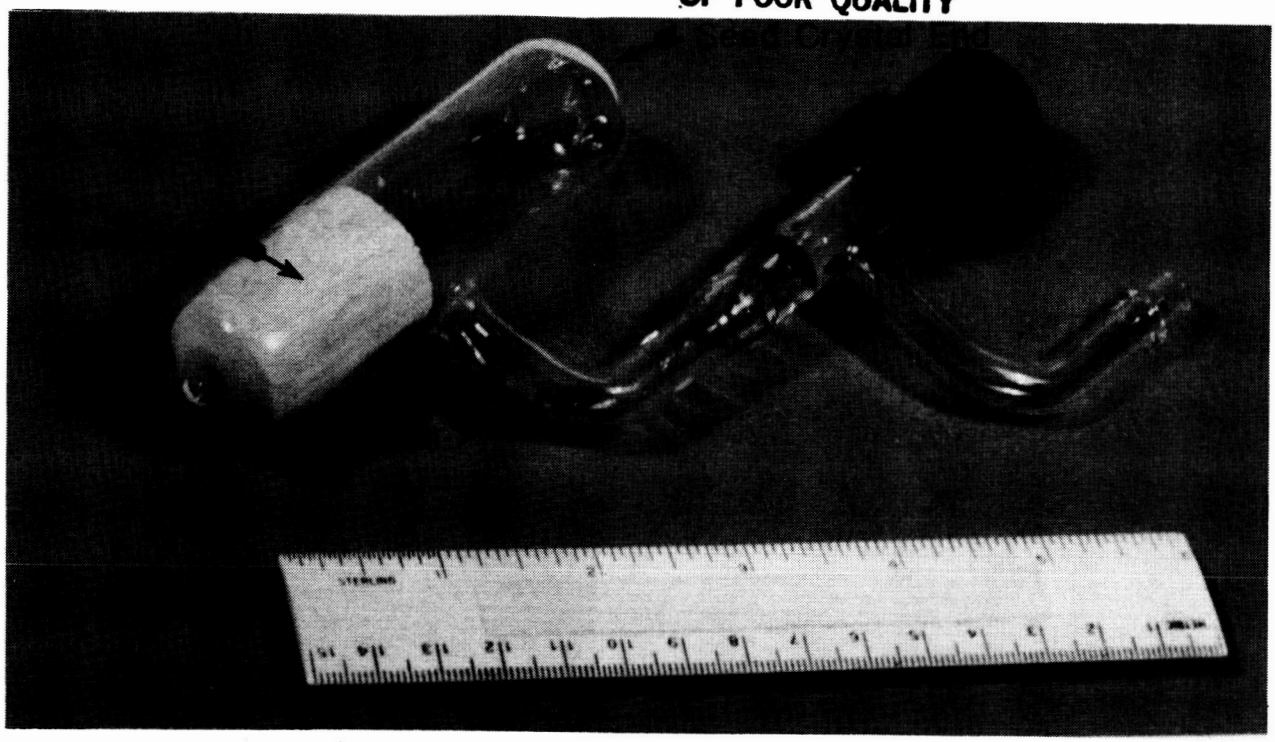

Figure 3 Monomer Crystallization Growth Chamber

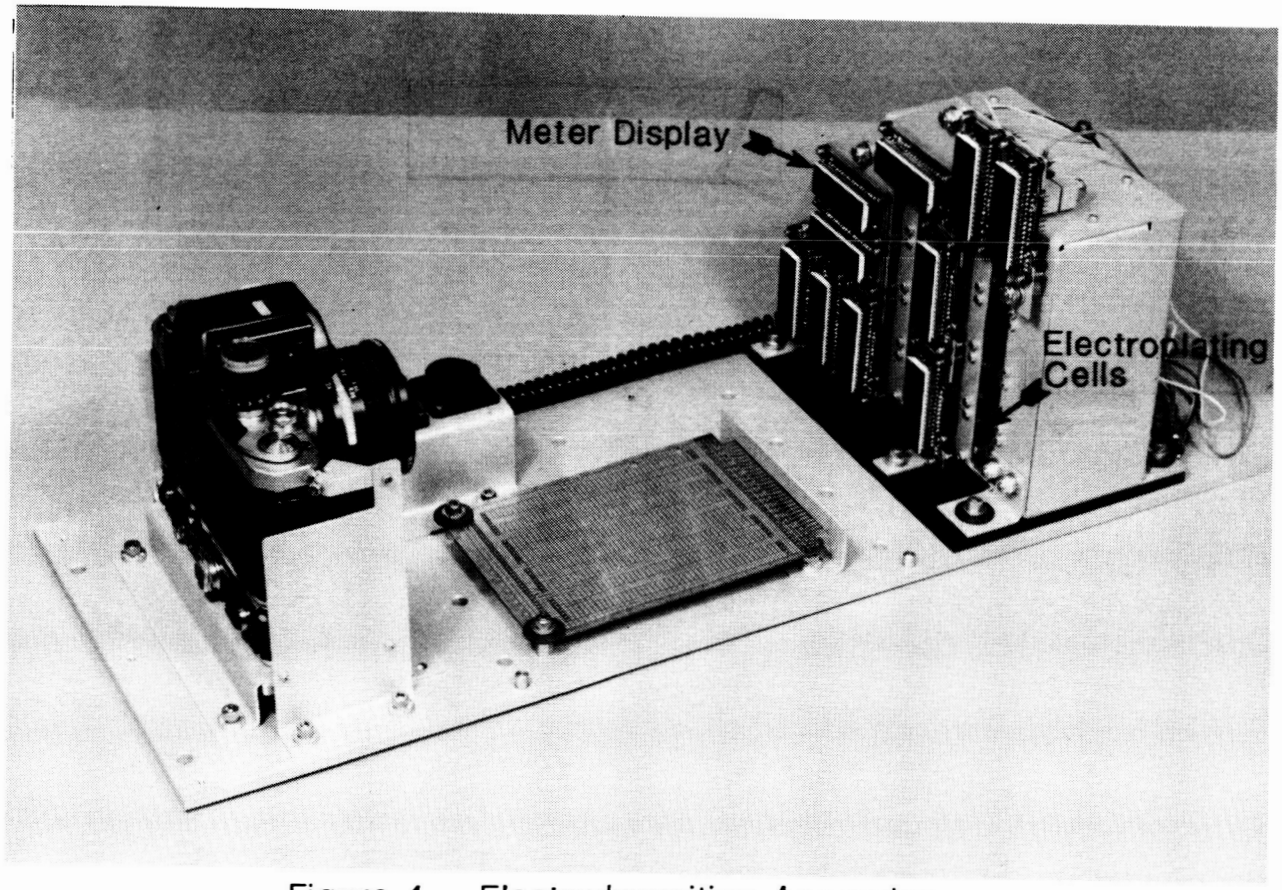

Figure 4 Electrodeposition Apparatus

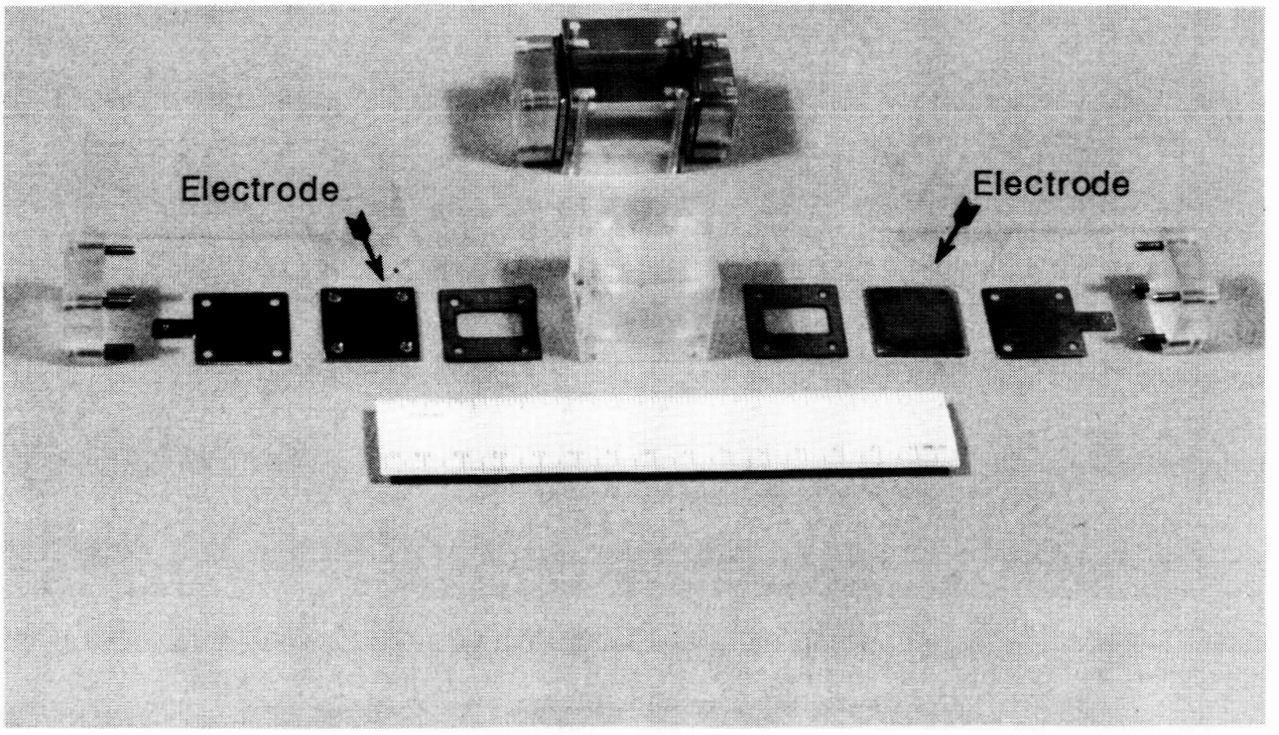

Figure 5 Electrodeposition Cell