JOURNAL OF SPACECRAFT AND ROCKETS
Vol. 31, No. 1, January–February 1994

Consort 3 Results

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Twelve experiments designed to obtain samples and data for a variety of microgravity materials processing investigations were carried on Consort 3, a sounding rocket flight. The overall purpose of the flight was to provide an opportunity to investigate the effects of microgravity on processes with potential for new technology development and commercial applications. The range of experimentation included investigations of immiscible polymer demixing rates, electrodeposition and electrocodeposition, elastomer-modified epoxy resins, foam formation, a number of biomaterial mixing processes, and polymer thin-film formation. Specific objectives and results are summarized for each experiment. The experiments were contained in a payload module which was launched and recovered within 4 h at the White Sands Missile Range in May, 1990. The microgravity portion of the flight lasted approximately 7 min. Apogee occurred 283 s after liftoff at an altitude of 189 statute miles. The integrated payload module configuration and mission sequence of events are briefly described. Evaluation of flight samples and recorded flight data is discussed for individual experiments. Consort 3 provided a large number of investigators with empirical information on processing materials in microgravity and on the effects of microgravity on the processes performed.

Introduction

CONSORT 3 was a continuation of low-gravity materials processing flights launched from White Sands Missile Range in New Mexico. The paper presents the payload module, the experiments, their objectives, and preliminary results. Further details of the payload module and experimental objectives for the Consort 3 mission are provided elsewhere.[1] Consort 3 was a reflight of Consort 2, which was aborted prior to achieving microgravity due to a rocket system failure. The objectives and results of its predecessor, Consort 1, are described elsewhere.[2,3] Consort 3 successfully flew on May 16, 1990, and provided just over 7 min of low gravity of approximately 100 μg. A Starfire 1 two-stage sounding rocket is used for Consort missions. The first stage is a TX664 solid rocket booster in a Terrier casing. The second stage is a Black Brant solid rocket motor. Telemetry, boost guidance, rate control, and recovery systems are also provided. The rocket is launched from a rail which is raised to a vertical position shortly before launch. This provides for late access to the payload, allowing experimenters to load sensitive biological specimens within a few hours of launch. The experiments and investigators are listed in Table 1.

Payload Module and Flight Operations

The integrated experiment payload module is shown in Fig. 1. The payload was approximately 3.6 m long, 0.44 m in diameter, and weighed approximately 280 kg. The lower two segments contained five experiments and two accelerometer systems that also flew on Consorts 1 and 2. Seven experiments were contained in the upper segment. Thus, a total of 12 different experimental apparatii flew on Consort 3.

The payload module was sealed by means of O-rings and hermetic connectors to maintain a pressure of approximately 1 atm throughout the flight. The experiments were mounted onto longerons attached to a bulkhead on one end and pinned on the other. This arrangement facilitates the integration of the experiments into the payload module, and permits an experiment to be assembled and tested on its own mounting plate. For launch operations, the payload was thermally conditioned to maintain an internal module temperature of $20 \pm 5°C$.

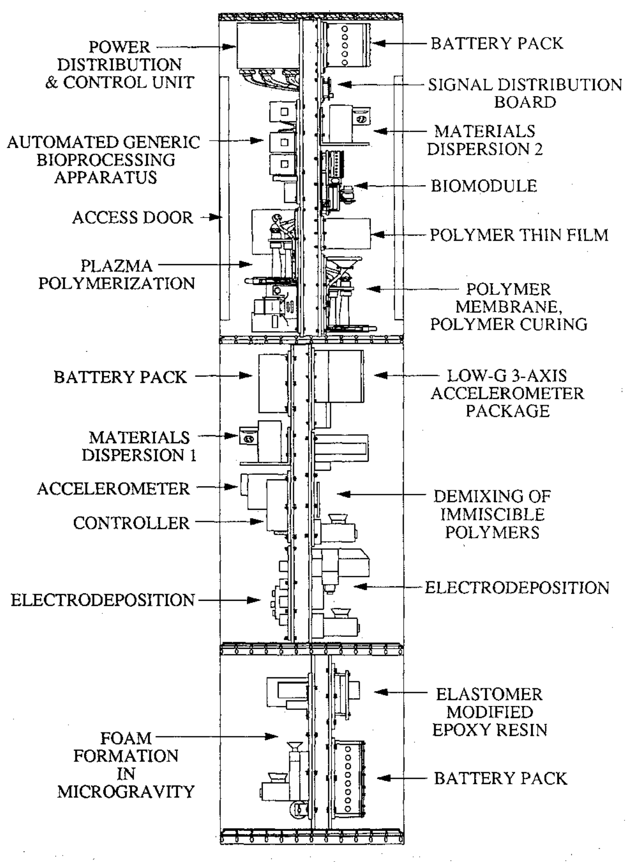

Fig. 1 Integrated experiment payload module.

Received Oct. 24, 1992; revision received Jan. 25, 1993; accepted for publication Feb. 2, 1993.

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Table 1 Consort 3 experiments

Experiment	Principal Investigator	Organization
1) Demixing of immiscible polymers	J.M. Van Alstine	University of Alabama in Huntsville (UAH)
2) Electrodeposition cells	C. Riley	UAH
	H. Abi-Akar	UAH
	G.W. Maybee	McDonnell Douglas Aerospace, Huntsville
3) Elastomer-modified epoxy resins	J.M. Harris	UAH
	F.C. Wessling	UAH
	J. Geibel	Consultant, Phillips Petroleum
4) Foam formation	S.P. McManus	UAH
	F.C. Wessling	UAH
	D. Lester	Thiokol Corporation
5) Flight experiment computer	C.C. Rupp	Marshall Amateur Radio Club
6) Accelerometers	J.A. Bijvoet	CMDS
7) Power distribution and control unit	C. Schwarz	MDA
8) Materials dispersion apparatus	J. Cassanto	Instrumentation Technology Associates
9) Automated generic bioprocessing apparatus	L. Stodieck	University of Colorado
	B. Spooner	Kansas State University
	J. Guelkema	Kansas State University
	P. Wong	Kansas State University
	M. Luttges	Bioserve Space Technologies
10) BioModule	R. Hammerstedt	Pennsylvania State University CCDS Center for Cell Research
11) Polymer thin film	D. Lester	Thiokol Corporation
12) Polymer membrane processing, multiphase polymer curing, and plasma polymerization	V. McGinniss	Battelle CCDS

The sequence of events for the Consort 3 flight is given in Table 2. Approximately 7.3 min of low gravity was attained. The payload was recovered from the desert floor, approximately 80 km from the launch complex, and returned within 4 h by helicopter to a launch complex facility for postflight processing.

Information on individual experiments that were flown is presented in the following material. The presentations are brief because of the large number of experiments and limited paper length.

Demixing of Immiscible Polymers

The Consort 3 experiments were designed in part to reconcile demixing time differences seen in previous low-gravity observations[4] from KC-135 aircraft and Shuttle flights STS-51D and STS-26. They were also intended to establish optimal systems for Joust, Get-Away-Special, and Spacehab experiments including the verification of aqueous polymer two-phase system emulsion kinetic demixing rates, and the effects of varying wall wetting, phase volume ratio, and chamber shape and volume on phase emulsion demixing rates. The rate of demixing is greatly influenced by the acceleration of gravity as well as by the ability of the liquids to wet the wall, and on liquid composition.

The latter studies were encompassed in a demixing of immiscible polymer (DIP) experiment consisting of a set of 14 quartz cuvettes filled with immiscible mixtures of polyethylene glycol (PEG) and water or dextran and water (see Fig. 2).[4,5] The cuvettes (4.2 ml cubes) were arranged in two rows of six cuvettes inside an adiabatic chamber. The cuvettes in the top row were coated with dextran and in the bottom row with PEG. External to each cuvette was a stirring motor with a magnet. A small stirring bar was inside each cuvette. Stirring was initiated well after the second stage separated from the payload and the rate control systems had nulled the payload body rates. The motor speed was decreased from that used on earlier flights to prevent the stirring bars from decoupling from the magnetic drive (with loss of mixing effiency).

Consort 3 provided an opportunity to evaluate magnetic mixing vs (potentially less reproducible) manual (ball shake) mixing used on STS-26.[5]

The stirring bars used on Consort 3 worked very well, but the mixing was less efficient than expected. The stirring bars do appear to provide a better view of the demixing process and ease of analysis. Figure 3 shows the demixing occurring 30 s after stirring.

The center of the photograph also shows two larger 25-ml chambers containing 14 ml of (7,5)I, 1/1 (V/V) system in chambers with preferred PEG-rich phase wetting (left side) and dextran-rich phase wetting (right side). These were shaped so that the geometry and surface tension would cause the two phases to separate. The only mixing of the material in these two chambers was due to accelerations and vibrations during launch. The chambers did not appear to readily demix, most probably due to phase viscosity and the surface-to-volume ratio and short flight time.

Although the Consort 3 flight did not allow enough time for the phase systems to completely demix, it did allow demixing to proceed for a longer time than on the KC-135. Photographs of the demixing phases were quantitatively analyzed as discussed previously.[4,5] The flight allowed us to reproduce kinetic demixing results seen on the Shuttle (see Table 3). In addition, it provided an opportunity to verify and test photographic and other apparatus approaches (including improved mixing regimes and techniques).

The rationale for the rocket flight was to support some unusual observations seen on STS-26 and STS-51D. These observations, described elsewhere,[4,5] pertained to two closely related phase systems which demixed very differently in low g. The two systems, (7,5)V and (7,5)I, differ only in salt composition and in the latter having an appreciable electrostatic

Table 2 Consort 3 sequence of events

Event	Time, s	Altitude, s.mi. MSL	Range, s.mi.	Velocity, ft/s	Q, psf	Flight path angle, deg
Max *Q*	7.0	1.89	0.11	1864	2987	83.77
BBVC	11.6	3.46	0.27	1675	1844	84.91
S19 End of guidance	18.0	5.78	0.43	2265	2265	85.76
Max *Q*—BBVC	21.2	7.27	0.52	2686	2413	85.77
BBVC burnout	43.0	27.08	2.07	7222	71	85.32
RCS arm	49.6	35.97	2.82	7037	15	85.12
Yo-yo despin	51.8	38.88	3.07	6968	9	85.07
Nose tip eject	54.4	42.28	3.36	6887	4	85.02
P/L motor sep & RCS enable	58.0	46.92	3.77	6774	1	84.93
Begin micro *g*	60.6	50.22	4.06	6693	0	84.82
Apogee	283.2	189.27	29.39	595	0	-0.04
End of micro *g*	500.8	56.81	52.26	6545	0	-85.44
RCS disable	509.0	46.51	53.04	6778	0	-85.74
RCS spin-up	511.0	43.94	53.23	6838	0	-85.76
S-19 power off	520.0	32.11	54.09	7084	400	-85.85
Max *Q* re-entry	525.0	25.38	54.57	7136	1059	-86.03
Main chute deployed	577.0	3.05	56.34	317	71	-71.20
Payload impact	844.0	0.93	55.68	21	0	-90.00

interfacial potential and droplet zeta potential. The first number refers to the weight percentage of dextran, the second to the weight percentage of polyethylene glycol (PG 8000) in water. The "V" refers to a buffer without zeta potential, and the "I" with zeta potential. The zeta potential apparently provides for colloidal stabilization since the (7,5)I system did not demix on STS-26. Interestingly enough, a (5,3.5)I system similar in zeta potential and other physical properties did readily demix on STS-26 and Consort 3. Since all three of these systems are candidates for carrying out cell separation in space, i.e., the (5,3.5)I was used on International Microgravity Lab (IML-1), we decided to attempt to reproduce the results. In addition, the rocket flight provided an opportunity to test a number of other observations of importance with regard to our programs. These included the effect of surface coatings on demixing and wall wetting and the effect of phase volume ratio on demixing rates.

Photographs were taken every 15 s. In many cases, the phase droplet regions grew to 4 mm in diameter during the first minute of demixing. Past that time droplet diameter is $>30\%$ of the cuvette width and the statistical certainty in the analysis falls off. Another problem with the analysis was the presence of air bubbles in the cuvettes. These bubbles were not present in our STS-26 experiments or in the Consort 3 cuvettes before launch. The bubbles appear to have resulted from temperature cycling of the rocket unit prior to liftoff.

In general, phase systems on Consort 3 behaved as they did on STS-26 and STS-51D with regard to the behavior of the three systems just discussed. In addition to a dependency of demixing rate on phase composition and resultant zeta potential, the importance of controlling phase wall wetting and phase volume ratio were verified. For example, a phase volume ratio of unity appears critical to maintaining optimal low *g* demixing rates. The (7,5)V and (5,3.5)I systems, but not the (7,5)I system, were used on Spacehab and IML-1.

The kinetic analysis gave good results for all but one of the ten chambers that yielded appreciable demixing (see Fig. 3). [This was the (5,3.5)I 6V/4V bottom row unit.] The linear regression for the plot of mean cell diameter vs time should yield a reasonably straight line with a y-axis intercept of about 0.1 to 0.2 mm. The log plot should yield a slope of about 1, consistent with emulsion demixing via the coalescence process. These were observed, and any slight differences in kinetics appear to be related to temperature, i.e., STS-26 at 28°C, Consort 3 at 20.5°C.

On STS-26 we saw some effect of coatings on demixing rates with the dextran-coated glass chambers exhibiting slower demixing than the PEG-wetting Plexiglas surfaces. Our analysis results suggest that the coatings did not greatly impact early demixing rates or appearance on Consort 3, although comparing chambers 2 to 3 and 5 to 11 does suggest 30% slower demixing in the dextran-coated chambers.

The coating result also supports the use of wetting materials such as Plexiglas and stainless steel, rather than coated glass, for the Spacehab apparatus chambers.

Electrodeposition

The electrodeposition experiment that flew on Consort 3 was configured to repeat the Consort 1 experiment with some changes in electrolytes, cathode materials, and hardware to improve experiment control and data acquisition. The major goal of this experiment was to reproduce the interesting results of the Consort 1 mission—specifically the high-rate nickel electrodeposition which produced a microcrystalline or amorphous form of the metal. Electrodeposition depends on gravity because of convective flow due to the depletion of ions in the electrolyte. Ten electrodeposition cells were utilized during the flight. Four of the cells were dedicated to electrocodeposition of inert particles into a metal matrix, four cells were used to study high-rate pure nickel deposition, and two cells were used to deposit cobalt at a high rate. Five cells were stirred, with four of those being codeposition cells and one containing neat nickel solution. The stirred nickel solution was included to determine if reduced gravity had any effect on a stirred metal deposition system. The codeposition cells consisted of two nickel/diamond cermets and two cobalt/chromium carbide cermets. The four codeposition cells were photographed

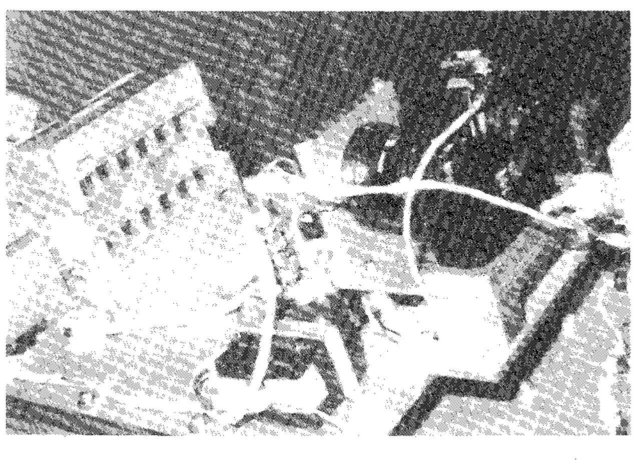

Fig. 2 Demixing of immiscible polymers experiment.

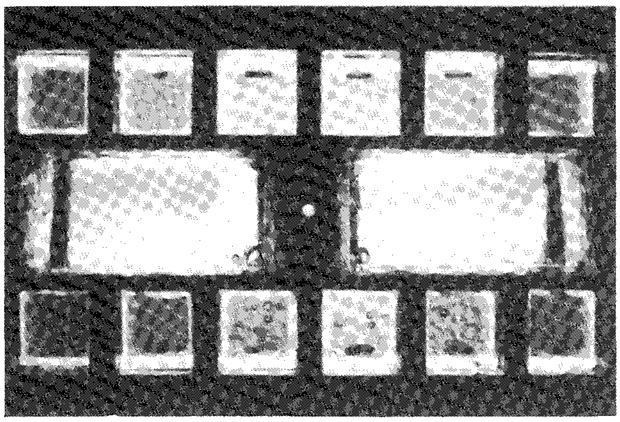

Fig. 3 Kinetic analysis—demixing.

periodically to monitor the suspension of the cermets during flight. All of the cells were controlled potentiostatically; cell current was allowed to float as required to maintain a constant cell voltage. The voltages were set to produce targeted equilibration currents as determined in 1 *g* during nonconvective operation.

The 7 min of reduced gravity resulted in very little codeposition, either for the diamond or chromium carbide. In fact, the larger (45 μm) diamond particles only left vacant tracts in the nickel deposited surface. They were not anchored deeply enough in the metal matrix to remain attached to the surface after the cessation of microgravity. This is opposed to the codeposition results of our Get-Away-Special, G-105, Shuttle experiment which showed excellent particle codeposition with the same cells that operated for 5 h. A small percentage of diamond particles on the order of 10 μm in diameter were codeposited uniformly in the nickel surface during the Consort 3 flight. The neat nickel cells all equilibrated at currents lower than the targeted current (80 ma/cm^2) except for one cell which used a glassy carbon cathode. Current in the cell with a glassy carbon cathode increased linearly from approximately 80 to 88 ma/cm^2 during the 7 min of operation. All other cells used gold-coated copper cathodes. X-ray diffraction of the nickel surfaces produced during the flight demonstrated only face-centered-cubic (FCC) structure, but the peaks were broader than those associated with FCC nickel deposited in 1 *g*. X-ray analysis of the two cobalt surfaces demonstrated that only the hexagonal-close-packed cobalt was deposited. Transmission electron microscopy studies of the nickel surfaces showed the presence of microcrystalline grains of nickel near the surface of the glassy carbon cathode which increased in size as the deposited layer thickened. Electron-diffraction patterns of the nickel surfaces deposited on either gold or glassy carbon showed only FCC nickel.

Elastomer-Modified Epoxy Resins

This experiment investigates the morphology and strength of elastomer-modified epoxy resins and can lead to better glues for manufacturing. The system selected for this experiment consists of three elastomer-modified epoxy resins. The elastomers are commercial products and are of the general class of materials called carboxy-terminated copolymers of butadiene and acrylonitrile (CTBN elastomers). These elastomers can be reacted with epoxy resins (typically a low-molecular-weight epoxy resin) to yield an epoxy-capped elastomer. Because the elastomer is capped by the epoxy resin, it can be cured just like a normal epoxy resin. However, because epoxy resins and the CTBN elastomers have different solubility parameters as the curing progresses, the driving forces for solubility decrease and the elastomer phase separates.[6] The curing process is slightly exothermic and the separated phases have different densities; thus, convective mixing may be influenced in the demixing process and this depends on gravity. The resultant deposition of rubber in epoxy will be determined by electron microscopy.[7]

The elastomer-modified epoxy resins experiment apparatus is essentially a flat-plate heater that heats a sample from both sides. The heater raised the sample temperature to 180°C on Consort 3 in approximately 90 s successfully. Crosslinking of the epoxy occurred. The glassy transition point changed from 46°C on the one-gravity samples to 52°C on the low-gravity samples.

Foam Formation Experiment

The foam formation experiment aboard the Consort 3 mission consisted of two piston and cylinder assemblies that contained chemical ingredients (polyol mixture with aluminum particulates and isocyanate) which were mixed to generate a rigid polyurethane foam. The isocyanate piston was actuated by nitrogen gas pressure to cause the isocyanate to flow into the cylinder containing polyol and aluminum particles. The two liquids were then mixed for 25 s by a motor-driven propeller. The mixed ingredients were then driven out of the chamber by the second nitrogen gas driven piston through a wire mesh funnel. Gravity affects the shape and size of bubbles of blowing agent entrapped in the polyurethane.

High pressure (~ 10 atm) of nitrogen gas was used to drive the isocyanate into the polyol. However, a gas pressure regulator lowered the pressure to approximately 5 atm for pushing the mixture through the exit funnel. The Consort 3 foam did not mix because of the failure of a seal. As a result, only the polyol and aluminum particles were ejected. However, photographs taken during low gravity showed uniform dispersion of the aluminum in the polyol.

The next three experiments, i.e., 1) materials dispersion apparatus, 2) automated generic bioprocessing apparatus, and 3) the BioModule, all intend to determine the effect of low gravity on biological processes on reasonably simple systems. Many Shuttle experiments have shown effects of low gravity on more complex systems such as man, dogs, monkeys, and fish.

Materials Dispersion Apparatus

There were two materials dispersion apparatus (MDAs) on Consort 3. Each MDA operates on the following principle:

Table 3 Phase demixing on STS-26 and Consort 3

System[a]	Vehicle	Mission	Demixing period, min	Regression parameters[b] C	D	γ	Averaged data points n
(7,5)V	Rocket	Consort 3	7	0.63	−0.61	0.99	4
(7,5)V	Space Shuttle	STS-26	120	0.66	−0.80	0.98	7
(5,3.5)I	Rocket	Consort 3	7	0.76	−0.70	0.98	4
(5,3.5)I	Space Shuttle	STS-26	120	0.51	−0.78	0.93	7

[a]As described in Refs. 4 and 5, values are for 4.2-ml systems in cubical chambers with PEG-rich phase preferring to wet the chamber wall.

[b]For equation $\log x = C \log \tau + D$ with $x =$ mm and $\tau =$ s; γ is the regression coefficient.

two blocks of inert material, with sample test wells facing each other in the upper and lower blocks, are held together under pressure with a sealing mechanism in an aerospace housing. The tests wells are misaligned at launch—thus separating the materials to be mixed. After microgravity has been achieved, the blocks are moved into alignment by means of a motor-cam mechanism allowing the wells to align. An option exists to mix a third or fourth material to fix a process while in the microgravity environment or prior to/during re-entry. Mixing occurs through the liquid-to-liquid diffusion process. Each MDA can have up to several dozen tests wells and each test well can accommodate samples in the 50 to 1000 m range.

Sixteen separate MDA experiments were flown on the Consort 3 mission; 46 samples on MDA-1 (rocket version/membrane casting), and 37 samples on MDA-2 (Shuttle version). Many experiments were run on both MDAs for redundancy. MDA-2 performed as expected, yielding 34 data points on 8 experiments. However, membrane coating and wetting studies on MDA-2 failed after microgravity due to fluid volatility and excessive heat from the slightly more than 1 h wait in the desert after impact. Other topics studied were collagen polymerization, fibrin clot formation, protein crystal stability, protein diffusion, zeolite crystals, baseline mixing, electro kinetic transport, and immiscible phases.

Zeolite crystal nucleation was performed in the MDA-2 unit. Zeolites may have commercial applications as filters and catalysts. For the first time ever, zeolites were successfully nucleated during space flight. After preliminary analyses, the space-grown zeolite nucleates looked different than expected from previous ground tests, when viewed and photographed hours after the microgravity environment of the rocket flight. The number of nucleates appears greater than in the ground controls. The nucleates include small cross-shaped crystals. Subsequent ground controls showed similar results, so the crystal formation may be related to the contacting method in the MDA rather than low gravity.

The other MDA unit (MDA-1) malfunctioned. It was dedicated to polymer membrane, other thin-film casting experiments, and basic fluid mixing. The unit is designed to cast membranes on 14 small 0.25-in.-diam glass disks slightly recessed in the top block of the unit and pressed in place. The blocks did not move to the microgravity position after the command was given. It is believed that the failure was due to one or more of these glass disks vibrating loose. The design has been modified to mechanically preclude this failure mode. These modifications have been tested in vibration and appear acceptable.

Although the MDA-1 unit malfunctioned, some of the experiments that did not require the fluids to contact and diffuse were a success. Delicate protein crystals were being tested with preservation techniques that provided 18 data points. From this flight, as well as the Consort 1 and 2 missions, new techniques have been developed to preserve protein crystals from the harsh re-entry *g* loads. Some protein crystals are fragile, and they fracture under acceleration loads around 12 *g*. Preservation techniques validated on Consort 3 allowed crystals to survive approximately 25 *g*. Protein crystal preservation is needed for orbital recovery capsule programs such as the recoverable, reusable satellite and the commercial experiment transporter.

Protein diffusion experiments on the Consort 3 sounding rocket flight studied the diffusion of two proteins: urokinase and horse myoglobin. Previous ground-control experiments using dyes had demonstrated that the mixing which occurred during sliding of the blocks was 0.37 + 0.16% of the original concentration. Diffusion analysis indicated that the amount of urokinase diffusion which could occur in the 6.6 min of microgravity (on Consort) permitted 0.64% of the urokinase to diffuse from the top chambers into the bottom when 0.25-in.-diam MDA wells were used. The protein diffusion experiments during the Consort 3 mission were designed to validate these estimates so that operational diffusion constants for different size proteins could be used to design functional experiments for protein crystal growth, biopolymer formation, and proteolytic enzyme activity.

Table 4 Thin films cured during microgravity

No. of films	Diameter of frame hole, in.	% formed of available frame size
6	0.25	100
3	0.50	60
2	0.75	50
1	1.00	50
0	1.50	0

The mixing experiments on Consort 3 showed the following results:

1) Mixing due to block sliding was 0.5% for 0.25-in. wells as compared to 0.3% for 0.125-in. wells.

2) Diffusion on Consort 3 was 0.65% for urokinase and 0.7% for myoglobin.

3) STS-37 fibrin lysis experiments showed complete lysis of fibrin clot by urokinase. Diffusion calculations predicted that only two-thirds of the clot should be dissolved.

4) Initial inspection of the wells during unloading of the fluids revealed that several of the urokinase wells in MDA-2 had become contaminated with methylene blue dye from adjacent wells. This cross contamination most probably occurred during removal of the top block from the bottom block. (This source of contamination was subsequently eliminated by modifying the unloading procedure.)

5) These were the first successful cross-diffusion experiments to be conducted in the MDAs on the Consort sounding rocket. However, the cross contamination just described interfered with the spectrophotometric measurements of the myoglobin. Therefore, multiple assays were conducted. The low volumes in the recovered wells made measurement of the myoglobin somewhat variable.

The results of this investigation enables experiments designed to investigate diffusion and proteolytic activity to proceed using the new configuration of the MDAs. New protein crystal growth experiments based on liquid-liquid diffusion and the osmotic dewatering method of Dr. Paul Todd can now be developed for longer-term Shuttle Middeck experiments since the initial mixing and protein diffusion rates can be estimated for the various MDA configurations, using the data obtained in these experiments.

Automated Generic Bioprocessing Apparatus

The automated generic bioprocessing apparatus (AGBA) was developed as a valuable tool to conduct a range of biomaterials processing experiments during reduced gravity. Configured for Consort 3, the AGBA weighed approximately 40 lb and occupied 1.1 ft^3. The payload consisted of six sets of polycarbonate blocks with misaligned wells containing sample materials. Experiments were initiated during reduced gravity by aligning sets of wells which allowed sample materials to mix through diffusion. In a similar manner, experiments were terminated at the end of the 7 min of reduced gravity by aligning the processed materials with wells containing fixative solutions. The AGBA supported 120 sets of wells with each individual well holding 0.2–0.8 ml.

The AGBA supported real-time acquisition and downlink of turbidity data from 32 reaction wells. In addition, temperature was monitored at three locations in the payload and controlled at 37°C in one of the six sets of polycarbonate blocks.

Principal investigators performed experiments on collagen polymerization, micro-organism metabolism, brine shrimp behavior, liposome formation, and clover rootlet inoculation. Results from these experiments were presented at the annual meeting of the American Society for Gravitational and Space Biology. In addition, results from the Consort 3 experiments were used by investigators to design experiments flown on the

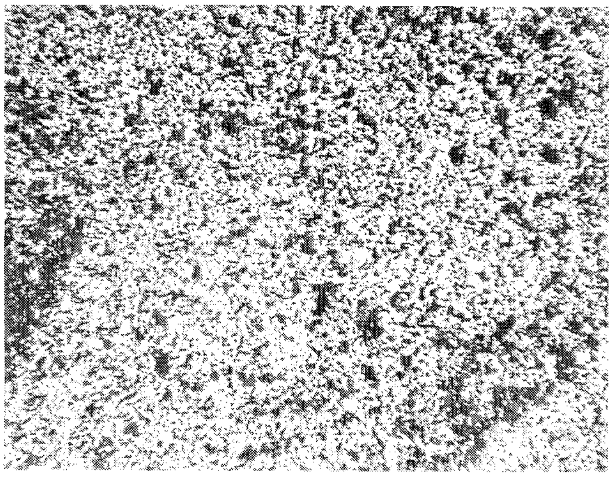

Fig. 4 40X photomicorgraph of free-standing thin film.

Space Shuttle in the BIMDA payload in April 1991 and again in August 1991. BIMDA employs technology similar to the AGBA in allowing diffusion-based mixing to initiate and terminate biomaterials processing experiments.

Two experiments were done on Consort 3 to characterize fluid mixing in the AGBA. The goal was to determine how much mixing resulted from diffusion and how much was due to convective flow induced by the well alignment process. Data from these experiments allowed investigators to better interpret results of experiments that were dependent on the quantity and quality of mixing.

In one case, 0.1% Trypan Blue in water was aligned with distilled water and a sucrose solution of density 1.2 g/ml. Mixing was quantified by measuring recovered sample optical density. In a second experiment, 0.1% Bromothymol Blue in a low-pH solution was aligned with high-pH solutions of water and a 1.2 g/ml sucrose solution. Color change at the liquid-liquid interface was monitored using the optical density measurement capability of the AGBA.

As expected, data showed that mixing was a strong function of fluid density and temperature. In addition, data revealed that mixing was much greater in reduced gravity than would have been predicted based on diffusion alone. Furthermore, mixing was found to be increased in reduced gravity compared to data from ground experiments. It appears that convective mixing is induced by the operation of the AGBA and that this effect is increased in reduced gravity. We have observed on the ground that disturbances at the liquid-liquid interface tend to stabilize or flatten following the completion of well alignment. In contrast, the interface remains sigmoidal following alignment as has been observed during reduced gravity on KC-135 flights.

Another experiment studied the effects of reduced gravity on pre-adult brine shrimp (*Artemia*). Experiments on this organism are geared toward 1) using Artemia as a staple system to understand the effects of gravity on growth and development, 2) producing food in space using Artemia as a candidate food source, and 3) using Artemia as a biomass of human sensitivity to pharmacological agents such as anesthetics.

For Consort 3, brine shrimp were hatched 24–48 h prior to launch. The shrimp were loaded into AGBA wells instrumented with optical density sensors. Spikes in the optical density signals were caused by shrimp crossing between the light source and sensor. Following the flight, optical density records were analyzed to infer shrimp swimming behavior during flight. Data from this experiment suggested swimming behavior was affected even in only 7 min of exposure to reduced gravity. Subsequent experiments on KC-135 flights have corroborated data from Consort 3.

Another investigation in the AGBA looked at mixed culture of algae-inducing *Chlorella* and *Euglena gracilis*. During reduced gravity, wells containing these organisms were aligned with those containing a variety of nutrient solutions. Just prior to the end of reduced gravity, the wells were aligned with a fixative solution. The number of organisms that crossed into the nutrient wells were counted and compared with ground controls. The objective was to provide information on the role of gravity in motility and chemolaxis. Data from this investigation were inconclusive. The nonmotile *Chlorella* mixed across the interface in a manner consistent with the fluids experiments just described. However, the data on the *Euglena* were not consistent across wells or experimental conditions. Future sounding rocket experimentation will be required to adequately understand these data.

Liposomes are vesicles assembled from lipid (fat) molecules. These spherical structures are valuable tools for analysis of membrane proteins and are currently being used as drug-delivery systems. Liposome assembly was accomplished in the AGBA during reduced gravity. Lipids were dissolved in solution with the aid of a mild detergent. During flight, wells containing the lipid solution were aligned with wells containing solution without detergent. As the detergent was diluted, liposomes could be formed.

Immediately following the flight, the processed materials were placed on electron microscopy (EM) grids and returned to the laboratory for EM analysis. Results showed a heterogeneous mix of liposomes which ranged in size from small to a few that were larger than any that had been produced on the ground under similar conditions. It was concluded that since the diffusion interface was relatively thin compared to the dimensions of the well, the large liposomes may have formed under reduced gravity and the small liposomes formed during re-entry and landing when considerable mixing would have taken place. This experiment has subsequently been repeated in the BIMDA payload which corroborated that liposomes formed in reduced gravity are much larger than those produced on the ground. Future experiments will focus on homogeneity, stability, and functional properties of space-made liposomes.

Yet another AGBA experiment evaluated the ability of nitrogen fixing bacteria to bind to clover seedlings, a necessary early step in root nodule formation. This process readily occurs in 1 *g* with certain types of plants called legumes. The root nodules fix nitrogen for these plants such that they can grow without the need for fertilizer. This process was investigated to determine if reduced gravity would interfere with nodule formation and as a first step to determine if reduced gravity can be used as a tool to induce nodule formation in nonlegumious plants such as wheat.

During the flight, cultures of nitrogen fixing bacteria were aligned with wells containing young clover sprouts. Prior to the end of reduced gravity, some of the sprouts were fixed

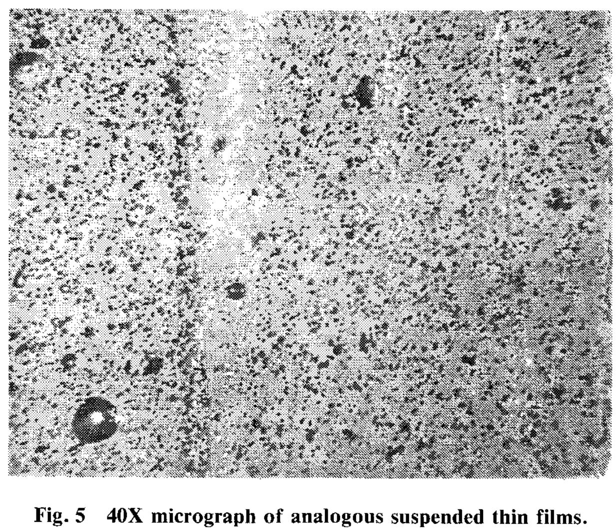

Fig. 5 40X micrograph of analogous suspended thin films.

with glutaraldehyde. These rootlets were sectioned and analyzed microscopically to quantify the binding of bacteria to the roots. Other rootlets were returned from flight unfixed and allowed to grow to determine if nodule formation occurred normally. Along with similar experiments done during KC-135 flights, results showed no effect of gravity on bacterial binding. Furthermore, nodule formation appeared to be unaffected by inoculation infection in reduced gravity.

Finally, experiments in the AGBA were conducted to study macromolecular assembly processes including collagen polymerization. Collagen is a prevalent protein in almost all tissues of the body. Fibers formed from collagen underlie the structure of tendons, bone, blood vessels, and even the cornea of the eye. If polymerized under the right conditions in reduced gravity, it may be possible to synthesize materials with properties similar to natural counterparts. Such materials could be used in biomedical transplant procedures.

During the flight of Consort 3, wells containing collagen dissolved in a low-concentration acetic acid solution were aligned with wells containing a buffered solution at a relatively high pH. By raising the pH of the collagen, polymerization was initiated. The collagen samples were fixed prior to the end of reduced gravity. Histological examination of the recovered samples showed that polymerization did occur during the flight. However, the process had not proceeded far enough in the 7 min of flight available to allow adequate comparison with ground samples. Collagen polymerization is being done on the Space Shuttle which will allow sufficient time for the process to go to completion. In addition, small forcing functions such as electric fields will be applied to align the collagen fibers during polymerization with the goal of producing highly organized materials.

BioModule

Effective biotechnology experiments in the microgravity environment require generic equipment suitable for a variety of cell biology and biotechnology projects. Equipment is being developed that: 1) is simple and easy to duplicate at low cost; 2) grows from the needs of life scientists, not traditional engineering approaches; 3) can be easily modified to satisfy specific scientific needs; 4) expands from a common theme of "fluid addition followed by timed observations of samples;" and 5) is adaptable to payload requirements for a variety of vehicles.

The main BioModule unit occupies a volume of less than 3 l, weighs less than 9 kg, and draws only power and liftoff signal from the payload systems. Thirty-two silicone "T" devices to hold cells (main body) and solutions (side bladders) are provided. The main body is isolated from the side bladders by mechanical pressure on the connecting tubes. On receipt of a preprogrammed signal from a dedicated local computer, the pressure is released and a solution is displaced from the side bladder into the main body. This format allows addition of either of two solutions (e.g., stimulant and fixative) to the cells at any time during the mission.

The majority (27 of 32) of the individual solenoid chambers on Consort 3 worked exactly as planned. Accumulation of launch site "grit and dirt" before launch in/on critical surfaces accounted for the failure of five solenoids. The defect in design was corrected for the Consort 4 flight. All settings and procedures for use of the BioModule have been validated and are ready for use in upcoming flights.

The question for test was "Will chameleon skin pieces, when challenged with sufficient hormone to cause a color change from brown to green in 75 s on Earth, also change color in 75 s in the microgravity environment?" Our interpretation must be cautious because the missing data points (due to solenoid firing problems) came at critical periods in the experiment. Based on visual and light microscopic analysis, we conclude that the skin did not change color to the extent expected from our previous ground-control experiments. Rather, we noted a partial color change for all of the samples treated with hormone, both in extent of color change for individual pieces and fraction of pieces that responded.

Reasons for these observations, other than a "real" effect of microgravity, could include incomplete mixing of stimulant and skin pieces under the microgravity conditions; or damage to cells during preparation and loading into the BioModule. However, on a later flight (Consort 4, November 16, 1991) all skin pieces changed as expected when stimulated within 2 min of attaining low gravity, but four out of six skins did not respond when stimulated after 5 min of low gravity. The difference may be explained by an improvement of technique between Consorts 3 and 4.

Studies in the tadpole system were conducted to determine if these cells, when placed in the BioModule, would retain structure over the experimental period. No attempt was made in this mission to cause any color change during flight. The value of the tadpole system is that a color change (i.e., melanocyte movement) can be induced by a physical stimulus (i.e., light flash). If the system can be used in the BioModule, we would have an alternative method (to chemical stimulus by hormones) to test for ability of melanocytes to respond in microgravity. Tissue samples containing photosensitive amphibian melanocytes were incubated in the Consort 3 flight BioModule to determine if they were robust enough to withstand the rigors of space flight. Based on the two samples flown, the melanocytes behaved in the expected manner, and it is concluded that these cells will provide a valuable model system for studying the influences of microgravity on intracellular particle translocation.

The studies constitute an important aspect of our "proof of principle" studies for the BioModule. The design is sound, tissues can be handled on site, and tantalizing data on effects of microgravity have been gathered.

Thin-Film Zero-*g* Experiment

The incorporation of metal powders into thin-film structures is of great interest for proposed applications in the space environment. Metal powders added to liquid thin films in the Earth's gravational field drain before they can be used or cured.[8] This phenomenon results from the difference in densities between metal powders and the liquid polymers. The density of most film-forming polymers ranges from 0.8 to 1.2 g/cm^3, whereas the densities of metal powders range from 2.69 g/cm^3 for aluminum, to as great as 19.32 g/cm^3 for gold. Forming and curing particulate-loaded film in a microgravity environment eliminates this effect. The objectives of this ex-

Table 5 Samples analyzed for transmission spectra

No.	Particulate	Description	Type gravity	Thickness, μm
1	No aluminum	Suspended	1 *g*	30
2	No aluminum	Formed over plate	1 *g*	80
3	Aluminum	Suspended	1 *g*	30
4	Aluminum	Formed over plate	1 *g*	100
5	Aluminum	Formed over plate	Zero *g*	40
6	Aluminum	Suspended[a]	Zero *g*	30

[a]The term "Suspended" in Table 5 implies that the film was drawn over an opening in a plate and is supported only at the opening's edge.

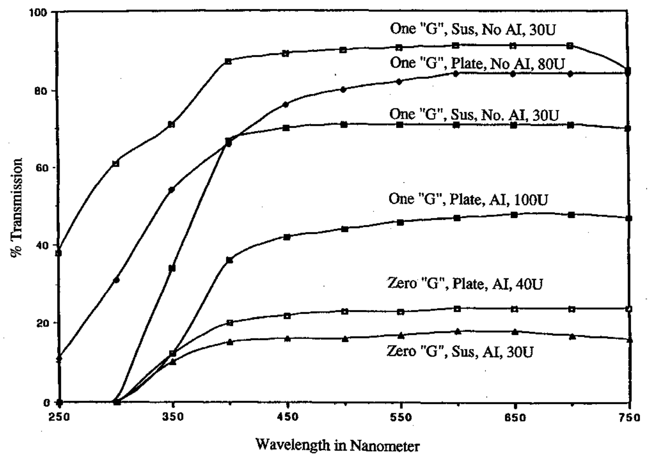

Fig. 6 Spectra of film found in Table 5.

periment were 1) form and cure liquid photocuring epoxy thin films containing flake aluminum during microgravity; and 2) characterize these films for morphology, light transmission, electrical conductivity, and strength. The characterized microgravity films are compared to analogous films formed in 1 *g*.

The experimental apparatus consists of a sealed reservoir containing a photocuring cycloaliphatic epoxide with 3% aluminum flake. Within the reservoir is found a mechanical stirring blade and perforated film-drawing plate. As microgravity was achieved, the mechanical stirrer was activated remixing the polymer and aluminum particles that might have separated during the spin-stabilized ascent. The film-drawing plate was then pulled from the reservoir-forming films that were cured in microgravity using two 6-W, short-wave, low-pressure mercury vapor, ultraviolet lamps.

The thin-film zero-*g* experiment is a self-contained experiment in that it includes its own power supply and event-sequencing device. Four 12-V, lead acid gel, Yuasa batteries provided the power for the experiment. The sequence of events is controlled by two Raymond model 1060, six single-pole double-throw timers. The timers were actuated during liftoff by a greater than 5 *g* vertical impulse.

The polymer-containment housing was removed from the thin-film zero-*g* experiment and that apparatus inspected shortly after the successful flight of Consort 3. This inspection showed that the experiment had performed as planned. The entire film-drawing plate was covered with a polymer-cured thin film. In addition, 12 of the 19 film frames contained free-standing thin films. The details of this inspection are contained in Table 4. Following this look, the apparatus with samples intact were packaged and shipped for an in-depth postflight analysis.

Thin films formed in microgravity are loaded with significantly more aluminum particulate than analogous films formed at 1 *g*. Figure 4 shows a 40X photomicrograph of a typical free-standing thin film formed in microgravity. This sample is a portion of a ¾-in.-diam film, and measures 28-μm thick. The photomicrograph was made using both reflected and transmitted light. The transmitted light was filtered blue, resulting in the blue background of the micrograph. The blue area of the micrograph corresponds to particulate free areas in the thin film.

Figure 5 shows a 40X micrograph of an analogous suspended thin film formed in 1 *g*. The film thickness is 29 μm. The aluminum concentration is higher in the film formed in microgravity. This is typical of all of the microgravity samples.

Examination of the films with a stereomicroscope shows the aluminum particles to be completely included within the thin film and not found on the surface. It was also observed that the planer orientation of the flake aluminum was random. This results in a film that reflects light in a diffuse manner. The flake aluminum has a particle size of approximately 5–30 μm on the large plane and a few microns thick. These dimensions allow free rotation of many aluminum particles within the fluid found between the surfaces of the thin films without interaction with surface formation forces.

These films are effectively nonconductive. Microscopic examination shows that the surface of the films is exclusively composed of polymer and that the flake aluminum particles found within the thin films are supported in a matrix of polymer that effectively isolates large numbers of aluminum particles from direct contact. The electric conductivity of these thin films is approximately that of the base polymer alone.

The percent of transmitted light through films formed in both microgravity and 1 *g* was measured from 750 to 250 nm on an ultraviolet, visible, and near infrared photospectrometer. Six samples were analyzed. A listing of these films is found in Table 5. The spectra of samples in Table 5 is shown in Fig. 6.

Microgravity-formed films consistently showed lower percentages of transmitted light when compared to analogous films formed at 1 *g*. These data quantify the obscuring effect of suspended aluminum flake found in the films. Particulate loading densities can be described as a function of transmitted light in the visible spectrum.

The tensile strength of six films was determined. No difference is observed between the strength of films formed in microgravity and 1 *g*.

Summary and Conclusions

The microgravity time available on Consort is sufficient to gain extensive data on materials processing. Five experiments which flew on Consort 1 flew again on Consort 3. These were modified to improve performance and/or to make changes in materials and protocols dictated by Consort 1 results. The demixing of immiscible polymers experiment photographed the initial stages of demixing, and some systems separated after their mixing in low gravity. This information will be used to improve biological separations on Spacehab. The elastomer-modified epoxy resins experiment yielded samples processed in low gravity that were adequate for determining the morphology of these resins, as well as provided specimens for tensile testing. The electrodeposition experiment obtained samples of nickel and cobalt deposited over a range of current densities and also obtained codeposited particles and metals. Analysis of nickel deposited at high current densities revealed a microcrystalline FCC structure rather than a true amorphous material. The experiment was reflown on Consort 4. A sample of polyurethane foam with aluminum particles was not made due to the failure of a seal. The MDA allows several researchers the opportunity to study a variety of phenomena including bone marrow studies, organic cell growth, and diffusion phenomena. The small test chambers allow many researchers to perform experiments in a small volume.

Four of the new experiments deal with polymers in low gravity. The thin-film zero-*g* experiment used ultraviolet light to cure a polymer liquid containing suspended aluminum flake. The resultant thin film was evaluated for the distribution of aluminum flakes and for optical and electrical properties. The investigation into polymer membrane processing sought to gain an understanding of the effect of convection-driven currents in the transport processes which occur during the evaporation casting of polymer membranes. The multiphase polymer curing experiment studied phase dispersion to acquire a better understanding of the fundamental properties of thermoplastic and thermosetting composites. The plasma polymerization in microgravity attempted to generate carbon particles or films from acetylene in a 10-kHz alternating current plasma.

The 12 experiments packaged in the Consort 3 payload returned a large amount of new data and significantly increased our understanding of many microgravity materials processes and possible commercial applications.

Acknowledgments

The authors gratefully acknowledge the support of the NASA Office of Commercial Programs for its support through Grant NAGW-812. Space Services Inc. (now a Division of EER Systems, Inc.) provided launch support and integrated the rocket with the payload module. McDonnell Douglas Aerospace, Huntsville, integrated the experiments into the payload module and supported launch operations. The Naval Air Warfare Center provided launch operations. The project was managed by the Consortium for Materials Development in Space at the University of Alabama in Huntsville.

The authors wish to thank the following persons who provided information for this paper: L. Stodieck, Bioserve Space Technologies, University of Colorado; R. Hammerstedt, Center for Cell Research, Pennsylvania State University; D. Lester, Thiokol Corporation; J. Cassanto, Instrumentation Technology Associates; and S. McManus, C. Riley, and J. Van Alstine, University of Alabama in Huntsville.

Payload trajectory and flight sequence data were provided by Space Services Inc., the launch services contractor for the Starfire sounding rocket.

Special thanks are due to the Marshall Amateur Radio Club and Charles C. Rupp for development of the experiment controller.

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