ments could also have been the cause of the higher density of primary dendrites in the latter specimens. In the specimen from Spacelab 1 only a few dendrites were observed in the regions with the three lowest solidification rates. At the highest rate, the number of primary dendrites was greater.

Follow-up analysis. The next analysis to be conducted will consist of measuring the sulfur, silicon, and phosphorus concentrations in the specimen. The results will be quantitatively combined with and related to those reported above, the measured temperatures and temperature gradient, and the rates of solidification (3).

Conclusions. The experiment on Spacelab 1 proceeded essentially as planned, but a number of unexpected phenomena occurred. The liquid metal column separated into two parts. Cavities were found at the ultimate position of the melting interface, which was closer to the specimen holder than programmed. Droplets of iron were observed on the surface of the Al<sub>2</sub>O<sub>3</sub> skin. This 50-µm-thick skin was rigid enough to keep the cast iron in its original shape during melting. The graphite structure did not clearly exhibit all five transitions in the solidification rate, contrary to observations in ground experiments. These results will be related in a quantitative way to the sulfur content as a function of position in the rod when the measurements are available.

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## **Tribology Experiment in Zero Gravity**

Abstract. A tribology experiment in zero gravity was performed during the orbital flight of Spacelab 1 to study the motion of liquid lubricants over solid surfaces. The absence of a significant gravitational force facilitates studies of the motion of liquid lubricants over solid surfaces as controlled by interfacial and capillary forces. Observations were made of phenomena associated with the liquid on one solid surface and also with the liquid between a pair of closely spaced surfaces. Typical photographic records obtained on Spacelab 1 are described.

The tribology experiment consisted of two parts, which were performed with separate experimental hardware and were designed to focus on different aspects of fluid-solid interactions. One part, referred to as the fluid wetting and spreading (FWS) study, was concerned with the motion of a drop of liquid lubricant on a solid surface under the influence of interfacial forces. The other part dealt with the morphology of a twophase film under shearing between the surfaces and is referred to as the journal bearing study because journal bearinglike devices were used to carry out the studies.

Fluid wetting and spreading. Although the tertiary equilibrium among gas, fluid, and solid phases is suitably described by the well-known formula of Young (1), the spreading process is inherently not an equilibrium phenomenon. The lowgravity conditions during the flight of Spacelab 1 provided a unique opportunity to observe the kinetics of wetting and spreading. In the FWS study, the combined influences of lubricant properties, surface chemistry, and topography were examined.

The FWS module is a mechanized fluid-dispensing device. A separate unit is used for each of four selected test fluids. Each module contains three geometrically identical surface specimens. Twelve fluid-surface combinations were used during the Spacelab 1 flight (2).

The study was conducted by photographing the wetting and spreading process as soon as the test fluid "surfaces" on the solid specimen. In each test sequence, approximately 24 µl of the test fluid was displaced through the central hole of each solid specimen. Filming started at 24 frames per second for 8 seconds, then changed to 1 frame per second for 8 minutes. All solid specimens were made of 440C stainless steel with various finishing and treatment conditions as indicated in (2). An oblique mirror beside each specimen showed the side profile.

Cinematographic records of wetting

and spreading of SRG-10 oil were analyzed by measuring the mean radius of the wetted spot at various times. Three phenomenological regimes were revealed by the spreading history:

1) Radius of each wetted spot grew to 0.15 cm in about 0.08 second with the same rate on all three specimens.

2) The wet radius on the barrier filmcoated specimen grew to 0.2 cm at the end of 0.8 second. The side profile was roughly hemispheric. Wet radii on the other two specimens increased to 0.3 cm in the same period.

3) The wet radius on the barrier filmcoated specimen stopped changing. Spreading continued on the other two specimens, with the wet radii reaching 0.50 and 0.38 cm, respectively, on the prewetted and clean specimens in 440 seconds. The spot shape on the prewetted specimen became oblong. A milli-g acceleration disturbance in this period has been established.

These results suggest that SRG-10 tends to wet and spread on a clean solid surface similar to 440C stainless steel, that the rate of advance of the contact line on a clean surface is very slow, and that barrier film coating can effectively prevent migration of the oil. Analysis of other FWS records is under way with the aid of a microdensitometer at Goddard Space Flight Center.

Journal bearing. In the normal functioning of an oil-lubricated journal bearing, the clearance space is only partially filled. On the unloaded side of the journal, natural drainage leaves behind a two-phase film which is connected to an outboard void space. Stable operation of a liquid-lubricated journal bearing is known to be dependent on the presence of the two-phase film. A series of journal bearing studies were performed on Spacelab 1 to examine the two-phase film in the capillary space of the journal bearing

The journal bearing module has a symmetrical, rigid rotor supported by a pair of experimental journal bearings. Three bearing configurations were used to control various kinematic and geometric parameters. In the first configuration, the bearings were of plain cylindrical geometry, and the rotor could be fitted with an unbalanced mass to yield an acceleration of 0.77 g. In the second, the bearings were shaped by three centrally preloaded arcs. In the third, the bearings were similar to those in the first configuration, but one end of the shaft was also fitted with a ball bearing to fix the operating eccentricity to three-fourths of the radial clearance at that end.

The experimental bearings are made of glass to permit viewing of the two-phase film. Inclined mirrors on either side of each bearing provide a full view. Encoding markers on the rotor are sensed optically to monitor speed and to furnish triggering signals for the camera and synchronized stroboscopic lighting.

The journal bearing module operates in the coast-down mode. The drive mechanism is disengaged when the rotor speed reaches 600 rev/min. The camera drive is synchronized with the rotor so that events related to unavoidable variations around the journal surface remain unchanged in successive cinematographic views. Noncontact proximity sensors along two mutually perpendicular axes are used to monitor the radial motion of the rotor.

The cinematographic records showed the following results, which correspond to the three bearing configurations described above:

1) With the balanced rotor, a streamer structure which almost wraps around the full circumference is seen. The appearance fluctuates and repeats in approximately every other frame. This suggests the presence of a one-half rotational rate oscillation. The possibility of such a flow structure was previously postulated (3). This general appearance continues as the rotor slows down to about 200 rev/min. The void content of the streamer structure is somewhat reduced at the lower speeds. With the unbalanced rotor, the two-phase morphology changes with speed. At the higher speeds, there is a more pronounced circumferential variation in the flow structure in each frame, but the frame-to-frame fluctuation is less apparent. When the rotor speed falls below about 300 rev/min, the general appearance reverts back to that seen for the balanced rotor.

2) Well-defined void regions are fixed to the three-arc geometry. Occasionally, small isolated voids are seen going around. There are no significant frameto-frame fluctuations.

3) The general appearance is similar to that in the second case, except that fixed void regions are less extensive and are not divided into three groups.

Data from the proximity sensors remain to be analyzed.

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  The lubricants used were SRG-10 superrefined paraffinic oil; Bray 815Z, a vacuum-distilled perfluoroalkyl polyether; Apiezon C, molecularly distilled paraffinic; and a 90:10 blend of fourand five-ring polyphenyl ethers. Finishes of the 440C stainless steel surfaces were ground (roughness 200 to 300 nm, finished by parallel grinding) or polished (roughness 25 to 50 nm, random texture). The surfaces were either clean, prewetted, or barrier film-coated; barrier film was a brush-applied fluoropolymer in solution with perfluorinated cyclic ether per military standard MIL-B-81744.
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- 4. This experiment is a collaborative effort between Columbia University and the Materials Processing Laboratory of NASA-Marshall Space Flight Center (NASA-MSFC). Keith Demorest was another principal investigator when planning for the experiment began; his premature departure is sadly noted. S. F. Murrary and S. Calabrese of Rensselaer Polytechnic Institute contributed significantly to the conceptual development of the experimental approach and to the preliminary feasibility assessment. Final hardware preparation to ensure flight worthiness was capably carried out by technical staff at NASA-MSFC under the leadership of R. Taylor and W. Wall.

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## Protein Single Crystal Growth Under Microgravity

Abstract. The preparation of suitably large protein single crystals is essentially the rate-determining step of protein x-ray structure determinations. Attempts to produce single crystals with two model compounds— $\beta$ -galactosidase and lysozyme—under conditions of microgravity were successful. Crystals formed by salting out from solutions kept free of convection were 27 and 1000 times larger in volume, respectively, than those produced in the same apparatus but exposed to terrestrial gravitation.

Solving the three-dimensional molecular structure of proteins by x-ray diffraction analysis helps in understanding their manifold functions (catalysis, transport, and supporting functions). An essential condition for such structural investigations is the availability of sufficiently large (about 1 mm in each dimension) and well-shaped single crystals. Especially disadvantageous for such protein crystal growth is the sudden formation of multiple seeds. Instead of a few large crystals, numerous small crystallites are formed which are useless for x-ray analysis. Experiments have shown that this effect is mainly due to convection, which can be almost completely suppressed by crystallization in gels. However, fragile protein crystals, expecially those growing as needles, break and are destroyed when they touch the tight network of the gel.

It seemed reasonable to look for conditions under which three solutions of



Fig. 1. Protein chamber with large single crysals of lysozyme (left side) and salt chamber (right side).

different densities ( $\rho_B < \rho_P < \rho_S$ )—a protein solution (P), a salt solution (S), and a gel-free buffer solution (B)-could be simultaneously and slowly combined without turbulence. Since B is the lightest of these components, such an experiment cannot be carried out by layering the solutions on top of each other. Conditions of microgravity were chosen in order to avoid differences in density  $(\rho_{\rm B} \sim \rho_{\rm P} \sim \rho_{\rm S} \sim 0 \text{ g/cm}^3)$ . In the experiment setup designed for microgravity, the protein and salt solutions are separated by a sliding device. In a second system of containers the buffer solution is kept. When all the containers have been filled without bubbles, they are closed to the outside by elastic membranes, which allow pressure compensation. When microgravity conditions have been attained, the sliding device is moved without vibration in order to connect the three different containers. Counterdiffusion of P and S across the buffer solution then takes place. The slowly diffusing salt ions cause the protein molecules to crystallize.

Since thermal changes could occur during this process and could lead to convection even in the absence of gravitational forces, preliminary short-term experiments (6 minutes of microgravity) were carried out with sounding rockets (European TEXUS program) in order to study the diffusion process with Schlieren optics. The high molecular weight protein  $\beta$ -galactosidase and the salt ammonium sulfate, both dissolved in M/30 phosphate buffer at *p*H 6.4, were used for testing. The experiments under microgravity, in contrast to those under