

# Space-Age Metals

## Freed from gravity, materials reveal their mysteries

By CORINNA WU

**G**ravity can be a real drag—especially to scientists trying to observe how liquid metals mix together (or fail to) and how they freeze into solids. Researchers would love to alloy gold with rhodium in order to produce high-temperature electrical contacts, for example. Others want to study the atom-to-atom interactions of cooling metals as they coalesce into the crystal structures that define their solid forms.

Unfortunately for these materials scientists, the interaction of heat and gravity in a sample of molten metal creates currents of rising and falling material, preventing some metals from alloying and overwhelming the subtle interactions between molecules of pure metals and metals that do mix well.

On NASA's space shuttle, however, where gravity is only one-millionth as strong as on Earth, these bothersome flows disappear. Gravity doesn't separate metals, and in droplets of liquid metal floating weightlessly, the atomic interactions that lead to a solid's microscopic structure are laid bare.

Accordingly, materials scientists have taken advantage of this unique environment. In various shuttle experiments over the past 3 years, including several with disappointing results, researchers have begun to sort out previously unexplored factors that determine how metals mix and crystallize. Some of the tests use organic substances as surrogates for metals.

At two meetings held at NASA's Marshall Space Flight Center in Huntsville, Ala., this summer, researchers discussed their recent Shuttle experiments. What they've learned may one day help improve the processing of metals on Earth.

**M**artin E. Glicksman, Matthew B. Koss, and their colleagues at Rensselaer Polytechnic Institute in Troy, N.Y., have been conducting experiments in microgravity to learn about dendrites: branching structures, somewhat like snowflakes, that form as a molten metal hardens into a solid. The size, shape, and orientation of the interlocking dendrites strongly influence the

properties of the final material (SN: 12/3/94, p. 375).

On Earth, "gravity really messes up the data," Glicksman says. The researchers can develop better models for dendritic growth by first understanding what happens without gravity-driven, or convective, currents to churn up the sample.

"By eliminating convective effects," he notes, "we can see dendritic growth as a pure diffusion process. Then, we can go back and check how good the [model] is." Scientists try to use models of dendritic growth to predict a metal's strength and malleability.

In their experiments, the researchers substitute two transparent organic substances—succinonitrile and pivalic acid—

for a wide range of metals. Succinonitrile coalesces into the atomic structure called a body-centered cubic, like that of iron, whereas pivalic acid takes on the face-centered cubic structure of metals that include aluminum and copper. Unlike the metals, the organic compounds melt at a low temperature—succinonitrile at 57°C and pivalic acid at 36°C—and are transparent, characteristics that allow the researchers to videotape the solidification as it occurs.

"It's unusual to have a transparent organic that solidifies like a metal," says Glicksman.

Earth's gravity, by causing convection currents, affects the shape and rate of dendritic growth of succinonitrile and pivalic acid crystals. The researchers hoped to see a clearer picture than ever before of crystal growth on the shuttle. The experiments consisted of placing 100 milliliters of each substance in a sealed chamber, keeping it at a precisely controlled temperature just below its melting point, then turning on two video cameras and stinging the liquid with a cool needle to start dendrite growth.

The Rensselaer team, controlling the apparatus from the ground, did hundreds of such experiments during three flights on the space shuttle Columbia. Two experiments using succinonitrile flew in 1994 and 1996 (SN: 3/16/96, p. 165). The most recent experiment, on pivalic acid, flew in late 1997. The experiments, revealing

### Gravity-defying golf

This could be NASA's biggest contribution to golf since Apollo 14 astronaut Alan Shepard launched a few shots on the moon in 1971. Earthbound golfers can now hit the links with clubs made from materials tested on the space shuttle.

This year, Liquidmetal Golf in Laguna Niguel, Calif., began producing clubs with heads made from a five-component metallic glass. Unlike conventional metals, the alloy doesn't have a rigid atomic structure. Instead, the combination of titanium, zirconium, nickel, beryllium, and copper is amorphous, its atoms randomly arranged.

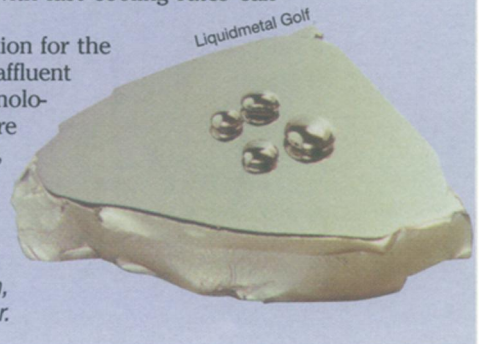
Golfers who have tried the clubs say that they combine hardness with a "soft feel," according to David S. Lee, head of manufacturing technology at Liquidmetal Golf. The material absorbs less energy than stainless steel and titanium—common materials for golf club heads—and so transfers more to the ball and less to the golfer's hands.

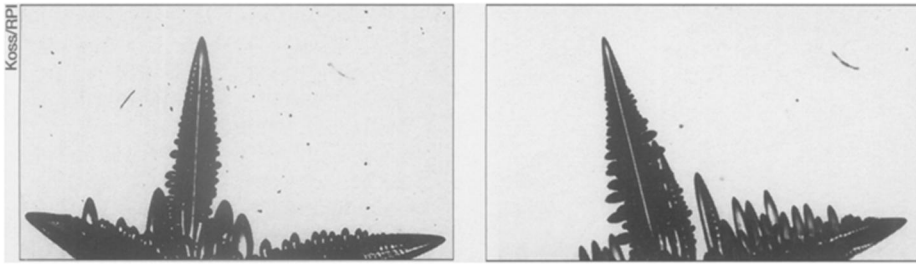
William L. Johnson and Atakan Peker of the California Institute of Technology in Pasadena invented the alloy in 1992. Samples of similar types of metallic glass from Caltech have flown on the space shuttle Columbia in TEMPUS, the containerless metal-processing device.

Certain combinations of metals form glasses easily when they are cooled rapidly, Lee says. This five-component alloy is "far and away the most forgiving" when it comes to commercial processing because it can be cooled more slowly than other metallic glasses. Slower cooling allows large batches of material time to turn to glass without crystallizing. Metallic glasses with fast cooling rates can only be made as powders or foils.

Golf clubs were an easy first application for the new glass because golfers are generally affluent and willing to embrace the newest technology, says Lee. He and his coworkers are looking at other uses for the material, especially in the aerospace, marine, and defense fields. —C.W.

Novel golf club heads are made from a metallic glass (right) containing titanium, zirconium, nickel, beryllium, and copper.





Subtle differences between pivalic acid crystals growing in space (left) and on Earth (right) can reveal how metals solidify. In space, the crystal grows 15 micrometers per second and acquires a blunt tip. On Earth, the crystal grows 35  $\mu\text{m}$  per second, it has a sharper tip, and the side branches lengthen more readily because gravity pulls heat away from the ends.

slower crystal growth than on Earth, yielded a wealth of information that the scientists are still sifting through, Glicksman says.

The pivalic acid formed some "surprising" dendritic shapes, he reports. Before the flight, other scientists had doubts about whether pivalic acid would freeze under weightless conditions as metals do. The successful crystallization appears to settle that argument. "It worked like a charm," says Glicksman.

**M**icrogravity may allow metals to do things they can't do at all on Earth. About 20 years ago, scientists started reexamining a group of metals known as immiscible alloys, says Barry Andrews of the University of Alabama at Birmingham. Like oil and water, these metal combinations simply don't mix well. Try to shake them up and they bead into droplets, eventually settling into two separate layers. Consequently, says Andrews, engineers have simply avoided combinations such as aluminum with indium, which could be used to make wear-resistant bearings.

Still, he continues, "they have the potential for some outstanding characteristics." If they could be thoroughly blended, these immiscible metals could yield new superconductors, magnetic materials, or catalysts, he speculates.

Early experiments conducted in low gravity—for example, in brief, high-altitude airplane flights (SN: 12/6/97, p. 362)—showed that molten, immiscible metals still separate when mixed together, but they do so in an unusual way. Instead of the denser metal settling to the bottom of the container, one component—and not necessarily the denser—migrates outward toward the container walls. The cooled, solidified samples look like hard-boiled eggs.

Without much gravity pulling one metal downward, the attractive force of the container walls takes over. The more adherent metal clings to the sides of the container like water sheeting over a pane of glass.

Andrews decided to look to space to examine this "wetting" behavior more closely. He chose to study the kinds of interactions that would occur in a mix-

ture of aluminum and indium. "We want to understand the physics behind its behavior" to better understand why some other metals won't mix, he says.

In collaboration with Sam R. Coriell of the National Institute of Standards and Technology in Gaithersburg, Md., Andrews designed another low-temperature experiment using organic molecules to model the metals. Astronauts on the late-1997 shuttle flight mixed the two transparent organic liquids, succinonitrile and glycerin, to represent aluminum and indium, respectively. The researchers wanted to see whether various combinations of the materials would be affected differently by the container walls.

Like their metal counterparts, the two substances are immiscible. The astronauts placed 12 samples with different

proportions of succinonitrile and glycerin between glass slides, then heated them and let them cool to see how they would behave.

Instead of examining samples after the substances had solidified, as in the earlier experiments with the metals themselves, the researchers included in the shuttle experiment a videocamera that taped the progress of the solidification. At a critical concentration, the videotape showed droplets of succinonitrile moving to and coating the container walls. "At 50 percent glycerin, it hit that magic transition for perfect wetting," Andrews says.

Whether these new results mean immiscible alloys could be mixed successfully in space or on Earth remains to be seen. "This is one of the first experiments on this effect," Andrews says. Ultimately, "we want to understand what's going on and maybe learn a few tricks so we can control the process."

Coriell, who does computer modeling of how metals freeze, and Andrews ran an experiment with actual metals in 1996 on Columbia. Using a furnace owned by the European Space Agency, astronauts heated and cooled three samples of aluminum and indium of different compositions at temperatures over 1,000°C. Preliminary experiments done in low gravity had shown that fibers of indium formed inside a matrix of aluminum.

The experiment "did not go as smoothly as we had hoped," says Andrews.



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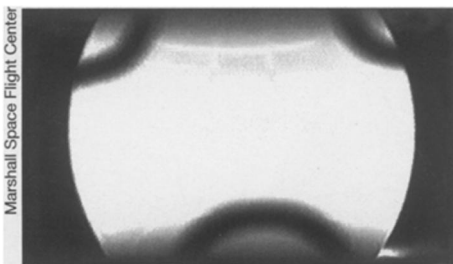
Some data were lost, and several samples formed bubbles. The researchers are still analyzing the unexpected imperfections.

**B**ased on earlier results, Andrews chose an aluminum nitride container for the 1996 experiments in order to minimize the wetting behavior. Some scientists, however, have gotten rid of containers altogether. An apparatus called the Electromagnetic Containerless Processing Facility (referred to by its German acronym TEMPUS) flew on two shuttle missions in 1994 and the summer of 1997.

Built for the German space agency, TEMPUS uses an electromagnetic field to levitate droplets of molten metal about 7 millimeters in diameter. Scientists can then heat and cool the samples and measure their physical properties.

"The sample is completely spherical. This is something that we can only achieve in microgravity," says project scientist Ivan Egly of the German Aerospace Center in Cologne. Spherical samples can provide more accurate measurements.

The containerless technique enables scientists to study crystallization over a greater temperature range than is possible on Earth. To study the crystallization,



A palladium-copper-silicon sphere glows when heated in an apparatus that holds metal samples electromagnetically.

the scientists melt the sample, then cool it carefully below its freezing point. Ordinarily, "any scratch on the container causes it to immediately solidify," says Jan R. Rogers, the U.S. project scientist for TEMPUS who is based at the Marshall Space Flight Center. The scratches serve as nucleation sites, or starting points for the crystal formation.

Without those imperfections, though, the molten metal globules can cool past the freezing point and remain liquid. Then, Rogers says, TEMPUS stabs the sample with a needle, triggering crystallization when the scientists are prepared to measure it.

On Earth, containerless processing devices need powerful magnetic fields. "Metals have a high density, so it takes a

lot of force to lift them up against gravity," Rogers says. Those high fields also tend to heat the samples, which then have to be cooled with a stream of gas, introducing impurities.

The first flight of TEMPUS was "not that successful," Rogers says, but the 1997 flight yielded much useful information. TEMPUS processed 18 samples, ranging from individual metals to alloys combining two to five elements. The remotely controlled apparatus measured the viscosity, surface tension, and other physical characteristics of the liquids at temperatures lower than could be achieved on Earth.

The basic information gleaned from these studies could help scientists develop better models to predict the properties of metals and metal alloys, says Coriell. Those, in turn, could lead toward ways to improve metal processing—increasing its efficiency, reducing its cost, or perhaps creating new alloys with interesting properties.

Could metal alloys be processed commercially in space, considering that microgravity allows them to mix in unusual ways? Sure, Andrews says, but "could anyone afford to purchase them? Probably not." It seems that even materials that are born in space will have to be manufactured on Earth. □

## Biochemistry

### New penicillin booby-traps bacteria

For years after its introduction as a drug in the 1940s, penicillin served as the first-line defense against bacterial infections. Bacteria evolved, however, into new strains with the ability to survive the drug. Now, many infections are resistant to penicillin and other antibiotics, fueling fears that doctors may soon run out of tools to keep these diseases in check.

A new study suggests that, with modification, penicillin might still have some punch left. Researchers at the University of Limerick in Ireland have attached a molecular booby trap to penicillin that can potentially defeat resistant bacteria. Timothy P. Smyth and his colleagues reported their strategy on Oct. 9 in the online version of the *JOURNAL OF ORGANIC CHEMISTRY*.

Bacterial strains resistant to penicillin have enzymes called beta-lactamases, which clip a crucial ring of the penicillin molecule, rendering it ineffective. "Over 190 of these enzymes have been identified so far, and the count is rising," Smyth says.

He and his colleagues chemically modified penicillin so that it releases a molecular fragment when a beta-lactamase cuts the ring. The fragment can be designed to kill bacteria. The most effective penicillin molecule they have synthesized to date kills *Escherichia coli* in the test tube, but only at high doses. Any bacteria that do not produce a beta-lactamase would be destroyed by the regular action of the antibiotic.

A strategy currently used to overcome resistant bacteria combines penicillin with compounds that block beta-lactamases, thus protecting the antibiotic. Bacteria, however, quickly develop beta-lactamases that don't bind those substances. To avoid that problem, the Limerick team uses the enzyme itself as a trigger to release and activate the lethal fragment, Smyth says.

Although the scheme looks promising, he adds, "there is some way to go yet to deliver a therapeutically useful drug." —C.W.

### A new angle on a blood-cell hormone

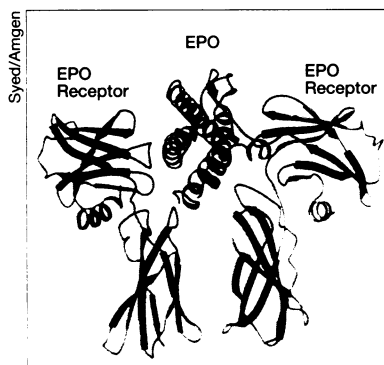
People who need a boost in red-blood-cell production—anaemic patients or those undergoing chemotherapy, for example—often take doses of the natural hormone erythropoietin (EPO). Now, a team of scientists in California has learned more about how EPO stimulates the creation of those cells.

Acting in the bone marrow, EPO binds simultaneously to two closely spaced molecules on the surface of a blood-precursor cell, thus triggering a cascade of biochemical reactions that transform the precursor cell into a red blood cell.

By looking at the three-dimensional structure of EPO with its bound receptor molecules, the researchers saw that the angle the receptors form is crucial. The receptors normally form a 120° angle, says Rashid S. Syed of Amgen in Thousand Oaks, Calif. This alignment best triggers the cell's biochemical cascade, he and his colleagues report in the Oct. 1 *NATURE*.

The three-dimensional structure seems to explain why smaller, synthetic proteins designed to bind to EPO receptors don't produce many red blood cells. These mimics move the receptors in-

to a less efficient, 180° angle and twist them slightly. Molecules that can correct both the angle and the twist might be better substitutes for EPO, Syed says. —C.W.



The three-dimensional structure of erythropoietin (center) bound to its two receptors (left and right).