## Nanolaminate Mirrors With "Piston" Figure-Control Actuators

Lightweight adaptive imaging mirrors can be built faster than can heavier glass mirrors.

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Efforts are under way to develop a special class of thin-shell curved mirrors for highresolution imaging in visible and infrared light in a variety of terrestrial or extraterrestrial applications. These mirrors can have diameters of the order of a meter and include metallic film reflectors on nanolaminate substrates supported by multiple distributed piezoceramic "piston"-type actuators for micron-level figure control. Whereas conventional glass mirrors of equivalent size and precision have areal mass densities between 50 and 150 kg/m<sup>2</sup>, the nanolaminate mirrors, including not only the reflector/shell portions but also the actuators and the backing structures needed to react the actuation forces, would have areal mass densities that may approach  $\approx 5 \text{ kg/m}^2$ . Moreover, whereas fabrication of a conventional glass mirror of equivalent precision takes several years, the reflector/shell portion of a nanolaminate mirror can be fabricated in less than a week, and its actuation system can be fabricated in 1 to 2 months.

The engineering of these mirrors involves a fusion of the technological heritage of multisegmented adaptive optics and deformable mirrors with more recent advances in metallic nanolaminates and in mathematical modeling of the deflections of thin, curved shells in response to displacements by multiple, distributed actuators. Because a nanolaminate shell is of the order of 10 times as strong as an otherwise identical shell made of a single, high-strength, non-nanolaminate metal suitable for mirror use, a nanolaminate mirror can be made very thin (typically between 100 and 150 µm from the back of the nanolaminate substrate to the front reflecting surface). The thinness and strength of the nanolaminate



A Cu/Cu<sub>x</sub>Zr<sub>v</sub> Nanolaminate was fabricated on a super-polished low-thermal-expansion glass form.

are what make it possible to use distributed "piston"-type actuators for surface figure control with minimal local concentrated distortion (called print-through in the art) at the actuation points.

Nanolaminate mirror substrates are fabricated in a direct replication process that consists of magnetron sputtering on precise, optical-quality master tools. As a result, the mirror substrates as manufactured (see figure) have nearly optical quality. Because nanolaminates are metals, their coefficients of thermal expansion are greater than those of the low-thermal-expansion glasses ordinarily used to make precise curved mirrors. Hence, backing structures should be made of materials with coefficients of thermal expansion matching those of the nanolaminate mirror shells. The actuators could be used to compensate for any residual thermally induced surface-figure distortions up to a few microns.

This work was done by Andrew Lowman, David Redding, Gregory Hickey, Jennifer Knight, Philip Moynihan, and Shyh-Shiuh Lih of Caltech and Troy Barbee of Lawrence Livermore National Laboratory for **NASA's Jet Propulsion Laboratory**. Further information is contained in a TSP [see page 1]. NPO-30222

## **Mixed Conducting Electrodes for Better AMTEC Cells**

These electrodes conduct both electrons and sodium cations.

Electrode materials that exhibit mixed conductivity (that is, both electronic and ionic conductivity) have been investigated in a continuing effort to improve the performance of the alkali metal thermal-to-electric converter (AMTEC). These electrode materials are intended primarily for use on the cathode side of the sodium-ion-conducting solid electrolyte of a sodium-based AMTEC cell. They may also prove useful in sodium-sulfur batteries, which are under study for use in electric vehicles.

An understanding of the roles played by the two types of conduction in the cathode of a sodium-based AMTEC cell is prerequisite to understanding the advantages afforded by these materials. In a sodiumbased AMTEC cell, the anode face of an anode/solid-electrolyte/cathode sandwich is exposed to Na vapor at a suitable pres-

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sure. Upon making contact with the solid electrolyte on the anode side, Na atoms oxidize to form Na<sup>+</sup> ions and electrons. Na<sup>+</sup> ions then travel through the electrolyte to the cathode. Na<sup>+</sup> ions leave the electrolyte at the cathode/electrolyte interface and are reduced by electrons that have been conducted through an external electrical load from the anode to the cathode. Once the Na<sup>+</sup> ions have been reduced to Na atoms, they travel through the cathode to vaporize into a volume where the Na vapor pressure is much lower than it is on the anode side. Thus, the cathode design is subject to competing requirements to be thin enough to allow transport of sodium to the lowpressure side, yet thick enough to afford adequate electronic conductivity.

The concept underlying the development of the present mixed conducting electrode materials is the following: The constraint on the thickness of the cathode can be eased by incorporating Na+-ionconducting material to facilitate transport of sodium through the cathode in ionic form. At the same time, by virtue of the electronically conducting material mixed with the ionically conducting material, reduction of Na+ ions to Na atoms can take place throughout the thickness of the cathode. The net effect is to reduce the diffusion and flow resistance to sodium through the electrode while reducing the electronic resistance by providing shorter conduction paths for electrons. Reduced resistance to both sodium transport and electronic conductivity results in an increase in electric power output.

Previous research had shown that mixed-conducting electrodes to improve the performance of an AMTEC cell could be made from mixtures of Mo (an electronic conductor) and Na<sub>2</sub>MoO<sub>4</sub> (an ionic conductor) or of W (an electronic conductor). Unfortunately, electrodes made of these mixtures do not last long: the vapor pressures of Na<sub>2</sub>MoO<sub>4</sub> and Na<sub>2</sub>WO<sub>4</sub> are so high at the typical operating temperature of a sodium AMTEC cell (between 1,020 and 1,120 K) that these ionically conduct-ing materials evaporate within a few hundred hours of operation.

The present mixed-conducting electrode materials are mixtures of Mo (as before, an electronic conductor) and  $Na_xTiO_2$  (a conductor of both electrons and  $Na^+$  ions).  $Na_xTiO_2$  can be formed by

exposing TiO<sub>2</sub> to Na vapor at a temperature >900 °C. Na, TiO<sub>2</sub> is a mixture of Na-Ti-O compounds, all of which are electronically conducting by virtue of the conductivity of TiO2, and ionically conducting toward Na<sup>+</sup> ions. In an experiment, an electrode made from a mixture of equal weight proportions of Mo and TiO<sub>2</sub> was found to perform well at a temperature of 830 °C (1,103 K) for >1,000 hours, with no significant change in either electrode power or transport properties. Moreover, the performance of this electrode was found to equal or exceed the performance of the best previously known AMTEC electrode, which was made of RhW.

This work was done by Margaret Ryan, Roger Williams, Margie Homer, and Liana Lara of Caltech for **NASA's Jet Propulsion Laboratory**. Further information is contained in a TSP [see page 1]. NPO-20920

# **Process for Encapsulating Protein Crystals**

Crystals can be grown in forms suitable for x-ray diffraction studies.

A process for growing protein crystals encapsulated within membranes has been invented. This process begins with the encapsulation of a nearly saturated aqueous protein solution inside semipermeable membranes to form microcapsules. The encapsulation is effected by use of special formulations of a dissolved protein and a surfactant in an aqueous first liquid phase, which is placed into contact with a second, immiscible liquid phase that contains one or more polymers that are insoluble in the first phase. The second phase becomes formed into the semipermeable membranes that surround microglobules of the first phase, thereby forming the microcapsules. Once formed, the microcapsules are then dehydrated osmotically by exposure to a concentrated salt or polymer solution. The dehydration forms supersaturated solutions inside the microcapsules, thereby enabling nucleation and growth of protein crystals inside the microcapsules.

By suitable formulation of the polymer or salt solution and of other physical and chemical parameters, one can control the rate of transport of water out of the microcapsules through the membranes and thereby create physicochemical conditions that favor the growth, within each microcapsule, of one or a few large crystals suitable for analysis by x-ray diffraction. The membrane polymer can be formulated to consist of low-molecular-weight molecules that do not interfere with the x-ray diffraction analysis of the encapsulated crystals. During dehydration, an electrostatic field can be applied to exert additional control over the rate of dehydration.

This protein-crystal-encapsulation process is expected to constitute the basis of protein-growth experiments to be performed on the space shuttle and the International Space Station. As envisioned, the experiments would involve the exposure of immiscible liquids to each other in sequences of steps under microgravitational conditions. The experiments are expected to contribute to knowledge of the precise conditions under which protein crystals form. By enhancing the ability to grow crystals suitable for x-ray diffraction analysis, this knowledge can be expected to benefit not only the space program but also medicine and the pharmaceutical industry.

The prior art in osmotic dehydration for growing protein crystals involves the use of a small chamber in which a planar reverseosmosis membrane is positioned between the mother liquor and a dehydrating salt solution. The prior art entails several disadvantages: (1) The nucleation and subsequent growth of protein crystals depend on

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increasing the concentration of precipitant and protein in the mother liquor; (2) there is no control over the effects of solute-driven convection on the surface of the crystal; (3) the crystals are not protected by any enclosure and thus are subject to physical damage as they are harvested and mounted; and (4) in some instances in the prior art, protein crystals have been protected by mounting them in aqueous gels, but this practice gives rise to the additional (as yet unsolved) problem of removing the gel material without adversely affecting the integrity of the protein crystals.

In contrast, the encapsulation of protein crystals in semipermeable membranes in the present process does not involve the use of gel, yet it creates closed environments that favor the growth of the crystals under prescribed conditions of controlled dehydration and protects the crystals against harsh environments that could otherwise damage the crystals.

In the present protein-crystal-encapsulation process, the microcapsules are spherical. The entire outer surface of the membrane of a microcapsule is accessible for osmotic dewatering as well as for and infiltration by hydrogen or hydroxyl ions. Such infiltration can be utilized to change the pH levels within microcapsules to favor or enhance protein saturation and subse-