

These **Temperature and Force Schedules** are followed in hot pressing of  $\text{La}_{3-x}\text{Te}_4$  or  $\text{La}_{3-x}\text{Yb}_x\text{Te}_4$  powder to consolidate it into a solid mass having superior thermoelectric properties. The force indicated here is calculated to yield a specified pressure when exerted over an area slightly more than 12 mm in diameter.

mass is sanded to remove graphite foil and boron nitride from its surface.

This work was done by Chen-Kuo Huang, Jean-Pierre Fleurial, G. Jeffrey Snyder, Richard Blair, and Andrew May of Caltech for NASA's Jet Propulsion Laboratory. Further information is contained in a TSP (see page 1).

In accordance with Public Law 96-517, the contractor has elected to retain title to this invention. Inquiries concerning rights for its commercial use should be addressed to:

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Refer to NPO-44356, volume and number of this NASA Tech Briefs issue, and the page number.

## Process for High-Rate Fabrication of Alumina Nanotemplates

Approximately regular hexagonal arrays of holes are formed in an anodizing process.

NASA's Jet Propulsion Laboratory, Pasadena, California

An anodizing process, at an early stage of development at the time of reporting the information for this article, has shown promise as a means of fabricating alumina nanotemplates integrated with silicon wafers. Alumina nanotemplates are basically layers of alumina, typically several microns thick, in which are formed approximately regular hexagonal arrays of holes having typical diameters of the order of 10 to 100 nm. Interest in alumina nanotemplates has grown in recent years because they have been found to be useful as templates in the fabrication of nanoscale magnetic, electronic, optoelectronic, and other devices. The present anodizing process is attractive for the fabrication of alumina nanotemplates integrated with silicon wafers in two respects: (1) the process involves self-ordering of the holes; that is, the holes as formed by the process are spontaneously arranged in approximately regular hexagonal arrays; and (2) the rates of growth (that is, elongation) of the holes are high enough to make the process compatible with other processes used in the mass production of integrated circuits.

In preparation for fabrication of alumina nanotemplates in this process, one first uses electron-beam evaporation to deposit thin films of titanium, followed by thin films of aluminum, on silicon

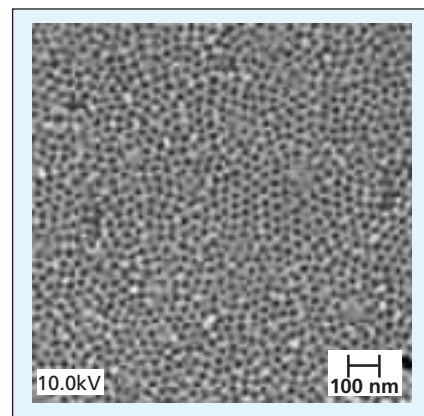
wafers. Then the alumina nanotemplates are formed by anodizing the aluminum layers, as described below.

In experiments in which the process was partially developed, the titanium films were 200 Å thick and the aluminum films were 5 µm thick. The aluminum films were oxidized to alumina, and the arrays of holes were formed by anodizing the aluminum in aqueous solutions of sulfuric and/or oxalic acid at room temperature (see figure). The diameters, spacings, and rates of growth of the holes were found to depend, variously, on the composition of the anodizing solution, the applied current, or the applied potential, as follows:

- In galvanostatically controlled anodizing, regardless of the chemical composition of the solution, relatively high current densities (50 to 100 mA/cm<sup>2</sup>) resulted in arrays of holes that were more nearly regular than were those formed at lower current densities.
- The rates of elongation of the holes were found to depend linearly on the applied current density: the observed factor of proportionality was 1.2 (µm/h)/(mA/cm<sup>2</sup>).
- For a given fixed current density and room temperature, the hole diameters were found to depend mainly on the chemical compositions of the anodizing

solutions. The holes produced in sulfuric acid solutions were smaller than those produced in oxalic acid solutions.

- The arrays of holes produced in sulfuric acid were more ordered than were those produced in oxalic acid.
- The breakdown voltage was found to decrease logarithmically with increasing concentration of sulfuric acid.
- The breakdown voltage was also found to decrease with temperature and to be accompanied by a decrease in hole diameter.
- The hole diameter was found to vary linearly with applied potential, with a



This **Alumina Nanotemplate** was made by room-temperature anodizing in an aqueous solution of 40 volume percent sulfuric acid at a current density of 50 mA/cm<sup>2</sup>. The hole diameter is 17 nm and the porosity is 16 percent.

slope of 2.1 nm/V. This slope differs from slopes (2.2 and 2.77 nm/V) reported for similar prior measurements on nanotemplates made from bulk aluminum. The differences among these slopes may be attributable to differences among impurities and defects in

bulk and electron-beam-evaporated aluminum specimens.

*This work was done by Nosang Myung, Jean-Pierre Fleurial, Minhee Yun, William West, and Daniel Choi of Caltech for NASA's Jet Propulsion Laboratory. Further information is contained in a TSP (see page 1).*

*This invention is owned by NASA, and a patent application has been filed. Inquiries concerning nonexclusive or exclusive license for its commercial development should be addressed to the Patent Counsel, NASA Management Office-JPL. Refer to NPO-40070.*

## Electroform/Plasma-Spray Laminates for X-Ray Optics

**Properties of lightweight components can be optimized.**

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Electroform/plasma-spray laminates have shown promise as lightweight, strong, low-thermal-expansion components for x-ray optics. The basic idea is to exploit both (1) the well-established art of fabrication of optical components by replication and (2) plasma spraying as a means of reinforcing a thin replica optic with one or more backing layer(s) having tailorable thermomechanical properties. In x-ray optics as in other applications, replication reduces the time and cost of fabrication because grinding and polishing can be limited to a few thick masters, from which many lightweight replicas can thereafter be made.

The first step in the fabrication of a component of the type in question is to make a replica optic by electroforming a thin layer of nickel on a master. Through proper control of the electro-

forming process conditions, it is possible to minimize residual stress and, hence, to minimize distortion in the replica. Next, a powder comprising ceramic particles coated with a metal compatible with the electroformed nickel is plasma-sprayed onto the backside of the nickel replica. Then through several repetitions and variations of the preceding steps or perhaps a small compressive stress, alternating layers of electroformed nickel and plasma-sprayed metal-coated ceramic powder are deposited.

The thicknesses of the layers and the composition of the metal-coated ceramic powder are chosen to optimize the strength, areal mass density, and toughness of the finished component. An important benefit of using both elec-

troforming and plasma spraying is the possibility of balancing stresses to a minimum level, which could be zero or perhaps a small net compressive stress designed to enhance the function of the component in its intended application.

*This work was done by Melville P. Ulmer, Michael Graham, and Semyon Vaynman of Northwestern University for Goddard Space Flight Center.*

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