Manufacturing & Prototyping

Electrochemical Hydrogen Peroxide Generator

Stable electrocatalysts can produce hydrogen peroxide under acidic conditions.

Lyndon B. Johnson Space Center, Houston, Texas

Two-electron reduction of oxygen to produce hydrogen peroxide is a much researched topic. Most of the work has been done in the production of hydrogen peroxide in basic media, in order to address the needs of the pulp and paper industry. However, peroxides under alkaline conditions show poor stabilities and are not useful in disinfection applications. There is a need to design electrocatalysts that are stable and provide good current and energy efficiencies to produce hydrogen peroxide under acidic conditions.

The innovation focuses on the *in situ* generation of hydrogen peroxide using an electrochemical cell having a gas diffusion electrode as the cathode (electrode connected to the negative pole of the power supply) and a platinized titanium anode. The cathode and anode compartments are separated by a readily available cation-exchange membrane (Nafion[®] 117). The anode compartment is fed with deionized water. Generation of oxygen is the anode reaction.

Protons from the anode compartment are transferred across the cation-exchange membrane to the cathode compartment by electrostatic attraction towards the negatively charged electrode. The cathode compartment is fed with oxygen. Here, hydrogen peroxide is generated by the reduction of oxygen. Water may also be generated in the cathode. A small amount of water is also transported across the membrane along with hydrated protons transported across the membrane. Generally, each proton is hydrated with 3–5 molecules.

The process is unique because hydrogen peroxide is formed as a high-purity aqueous solution. Since there are no hazardous chemicals or liquids used in the process, the disinfection product can be applied directly to water, before entering a water filtration unit to disinfect the incoming water and to prevent the build up of heterotrophic bacteria, for example, in carbon based filters.

The competitive advantages of this process are:

- 1. No consumable chemicals are needed in the process. The only raw materials needed are water and oxygen or air.
- 2. The product is pure and can therefore be used in disinfection applications directly or after proper dilution with water.
- 3. Oxygen generated in the anode compartment is used in the electrochemical reduction process; in addition, external oxygen is used to establish a high flow rate in the cathode compartment to remove the desired product efficiently. Exiting oxygen can be recycled after separation of liquid hydrogen peroxide product, if so desired.
- 4. The process can be designed for peroxide generation under microgravity conditions.
- 5. High concentrations of the order of 6-7 wt% can be generated by this

method. This method at the time of this reporting is superior to what other researchers have reported.

- 6. The cell design allows for stacking of cells to increase the hydrogen peroxide production.
- 7. The catalyst mix containing a diquaternary ammonium compound enabled not only higher concentration of hydrogen peroxide but also higher current efficiency, improved energy efficiency, and catalyst stability.
- 8. The activity of the catalyst is maintained even after repeated periods of system shutdown.
- 9. The catalyst system can be extended for fuel-cell cathodes with suitable modifications.

This work was done by Charles L.K. Tennakoon, Waheguru Singh, Kelvin C. Anderson, and Thomas Kinney of Lynntech, Inc. for Johnson Space Center.

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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Fabrication of Single, Vertically Aligned Carbon Nanotubes in 3D Nanoscale Architectures

Potential applications of this process are integrated circuits, nano switches, and biological sensors.

NASA's Jet Propulsion Laboratory, Pasadena, California

Plasma-enhanced chemical vapor deposition (PECVD) and high-throughput manufacturing techniques for integrating single, aligned carbon nanotubes (CNTs) into novel 3D nanoscale architectures have been developed. First, the PECVD growth technique ensures excellent alignment of the tubes, since the tubes align in the direction of the electric field in the plasma as they are growing. Second, the tubes generated with this technique are all metallic, so their chirality is predetermined, which is important for electronic applications. Third, a wafer-scale manufacturing process was developed that is high-throughput and low-cost, and yet enables the integration of just single, aligned tubes with nanoscale 3D architectures with unprecedented placement accuracy and does not rely on e-beam lithography. Such techniques should lend



(a) In the dc PECVD Growth Chamber, the sample was placed on a 3-in. (7.6-cm) Mo ring, where the wafer was transported from the load lock to the main chamber. (b) A single, vertically aligned tube is seen centered precisely within deep trenches, which was formed using high throughput processes.

themselves to the integration of PECVDgrown tubes for applications ranging from interconnects, nanoelectromechanical systems (NEMS), sensors, bioprobes, or other 3D electronic devices.

Chemically amplified polyhydroxystyrene-resin-based deep UV resists were used in conjunction with excimer laser-based ($\lambda = 248$ nm) step-and-repeat lithography to form Ni catalyst dots \approx 300 nm in diameter that nucleated single, vertically aligned tubes with high yield using dc PECVD growth. This is the first time such chemically amplified resists have been used, resulting in the nucleation of single, vertically aligned tubes.

In addition, novel 3D nanoscale architectures have been created using topdown techniques that integrate single,

vertically aligned tubes. These were enabled by implementing techniques that use deep-UV chemically amplified resists for small-feature-size resolution; optical lithography units that allow unprecedented control over layer-to-layer registration; and ICP (inductively coupled plasma) etching techniques that result in near-vertical, high-aspect-ratio, 3D nanoscale architectures, in conjunction with the use of materials that are structurally and chemically compatible with the high-temperature synthesis of the PECVD-grown tubes. The techniques offer a wafer-scale process solution for integrating single PECVD-grown nanotubes into novel architectures that should accelerate their integration in 3D electronics in general.

NASA can directly benefit from this technology for its extreme-environment planetary missions. Current Si transistors are inherently more susceptible to high radiation, and do not tolerate extremes in temperature. These novel 3D nanoscale architectures can form the basis for NEMS switches that are inherently less susceptible to radiation or to thermal extremes.

This work was done by Anupama B. Kaul, Krikor G. Megerian, Paul A. Von Allmen, and Richard L. Baron of Caltech for NASA's Jet Propulsion Laboratory. For more information, contact iaoffice@jpl.nasa.gov.

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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Process To Create High-Fidelity Lunar Dust Simulants

Marshall Space Flight Center, Alabama

A method was developed to create high-fidelity lunar dust simulants that better match the unique properties of lunar dust than the existing simulants. The new dust simulant is designed to more closely approximate the size, morphology, composition, and other important properties of lunar dust (including the presence of nanophase iron). A two-step process is required to create this dust simulant. The first step is to prepare a feedstock material that contains a high percentage of agglutinatelike particles with iron globules (including nanophase iron). The raw material selected must have the proper mineralogical composition. In the second processing step, the feedstock material from the first step is jet-milled to reduce the particle size to a range consistent with lunar dust.

This work was done by Robert Gustafson of Orbital Technologies Corp. for Marshall Space Flight Center. For more information, contact Sammy Nabors, MSFC Commercialization Assistance Lead, at sammy.a.nabors@nasa.gov. Refer to MFS-32729-1.