Materials & Coatings

Measuring Moisture Levels in Graphite Epoxy Composite Sandwich Structures

John F. Kennedy Space Center, Florida

Graphite epoxy composite (GEC) materials are used in the construction of rocket fairings, nose cones, interstage adapters, and heat shields due to their high strength and light weight. However, they absorb moisture depending on the environmental conditions they are exposed to prior to launch. Too much moisture absorption can become a problem when temperature and pressure changes experienced during launch cause the water to vaporize. The rapid state change of the water can result in structural failure of the material. In addition, heat and moisture combine to weaken GEC structures. Diffusion models that predict the total accumulated moisture content based on the environmental conditions are one accepted method of determining if the material strength has been reduced to an unacceptable level. However, there currently doesn't exist any field measurement technique to estimate the actual moisture content of a composite structure.

A multi-layer diffusion model was constructed with Mathematica to predict moisture absorption and desorption from the GEC sandwich structure. This model is used in conjunction with relative humidity/temperature sensors both on the inside and outside of the material to determine the moisture levels in the structure. Because the core materials have much higher diffusivity than the face sheets, a single relative humidity measurement will accurately reflect the moisture levels in the core. When combined with an external relative humidity measurement, the model can be used to determine the moisture levels in the face sheets. Since diffusion is temperaturedependent, the temperature measurements are used to determine the diffusivity of the face sheets for the model computations.

This work was done by Mark Nurge, Robert Youngquist, and Stanley Starr of Kennedy Space Center. Further information is contained in a TSP (see page 1). KSC-13499

Marshall Convergent Spray Formulation Improvement for High Temperatures

Lyndon B. Johnson Space Center, Houston, Texas

The Marshall Convergent Coating-1 (MCC-1) formulation was produced in the 1990s, and uses a standard bisphenol A epoxy resin system with a triamine accelerator. With the increasing heat rates forecast for the next generation of vehicles, higher-temperature sprayable coatings are needed.

This work substitutes the low-temperature epoxy resins used in the MCC-1 coating with epoxy phenolic, epoxy novalac, or resorcinolinic resins (higher carbon content), which will produce a higher char yield upon exposure to high heat and increased glass transition temperature.

High-temperature filler materials, such as granular cork and glass ecospheres, are also incorporated as part of the convergent spray process, but other sacrificial (ablative) materials are possible. In addition, the use of polyhedral oligomeric silsesquioxanes (POSS) nanoparticle hybrids will increase both reinforcement aspects and contribute to creating a tougher silacious char, which will reduce recession at higher heat rates. Use of expanding epoxy resin (lightweight MCC) systems are also useful in that they reduce system weight, have greater insulative properties, and a decrease in application times can be realized.

This work was done by Jack Scarpa and Chat Patterson of United Space Alliance for Johnson Space Center. For further information, contact the JSC Innovation Partnerships Office at (281) 483-3809. MSC-24644-1

Real-Time Deposition Monitor for Ultrathin Conductive Films

John H. Glenn Research Center, Cleveland, Ohio

A device has been developed that can be used for the real-time monitoring of ultrathin (2 Å or more) conductive films. The device responds in less than two microseconds, and can be used to monitor film depositions up to about 60 Å thick. Actual thickness monitoring capability will vary based on properties of the film being deposited. This is a single-use device, which, due to the very low device cost, can be disposable.

Conventional quartz/crystal microbalance devices have proven inadequate to monitor the thickness of Pd films during deposition of ultrathin films for hydrogen sensor devices. When the deposited film is less than 100 Å, the QCM measurements are inadequate to allow monitoring of the ultrathin films being developed. Thus, an improved, highsensitivity, real-time deposition monitor was needed to continue Pd film deposition development.

The new deposition monitor utilizes a surface acoustic wave (SAW) device in a differential delay-line configuration to produce both a reference response and a response for the portion of the device on which the film is being deposited. Both responses are monitored simultaneously during deposition. The reference response remains unchanged, while the attenuation of the sensing path (where the film is being deposited) varies as the film thickness increases.

This device utilizes the fact that on high-coupling piezoelectric substrates,

the attenuation of an SAW undergoes a transition from low to very high, and back to low as the conductivity of a film on the device surface goes from nonconductive to highly conductive. Thus, the sensing path response starts with a low insertion loss, and as a conductive film is deposited, the film conductivity increases, causing the device insertion loss to increase dramatically (by up to 80 dB or more), and then with continued film thickness increases (and the corresponding conductivity increases), the device insertion loss goes back down to the low level at which it started. This provides a continuous, real-time monitoring of film deposition. For use with different films, the device would need to be calibrated to provide an understanding of how film thickness is related to film conductivity, as the device is responding primarily to conductivity effects (and not to mass loading effects) in this ultrathin film regime.

This work was done by Jacqueline Hines of Applied Sensor Research & Development Corp. for Glenn Research Center. Further information is contained in a TSP (see page 1).

Inquiries concerning rights for the commercial use of this invention should be addressed to NASA Glenn Research Center, Innovative Partnerships Office, Attn: Steven Fedor, Mail Stop 4–8, 21000 Brookpark Road, Cleveland, Ohio 44135. Refer to LEW-18651-1.

Optimized Li-Ion Electrolytes Containing Triphenyl Phosphate as a Flame-Retardant Additive

This technology is applicable to portable electronics, electric vehicles, and other applications requiring safety and performance over a wide temperature range.

NASA's Jet Propulsion Laboratory, Pasadena, California

A number of future NASA missions involving the exploration of the Moon and Mars will be "human-rated" and thus require high-specific-energy rechargeable batteries that possess enhanced safety characteristics. Given that Li-ion technology is the most viable rechargeable energy storage device for near-term applications, effort has been devoted to improving the safety characteristics of this system. There is also a strong desire to develop Li-ion batteries with improved safety characteristics for terrestrial applications, most notably for hybrid electric vehicle (HEV) and plug-in hybrid electric vehicle (PHEV) automotive applications. Therefore, extensive effort has been devoted recently to developing non-flammable electrolytes to reduce the flammability of the cells/battery.

A number of electrolyte formulations have been developed, including systems that (1) incorporate greater concentrations of the flame-retardant additive (FRA); (2) use di-2,2,2-trifluoroethyl carbonate (DTFEC) as a co-solvent; (3) use 2,2,2-trifluoroethyl methyl carbonate (TFEMC); (4) use mono-fluoroethylene carbonate (FEC) as a co-solvent and/or a replacement for ethylene carbonate in the electrolyte mixture; and (5) utilize vinylene carbonate as a "SEI promoting" electrolyte additive, to build on the favorable results previously obtained.

To extend the family of electrolytes developed under previous work, a number of additional electrolyte formulations containing FRAs, most notably triphenyl phosphate (TPP), were investigated and demonstrated in experimental MCMB (mesocarbon microbeads) carbon-LiNi_{0.8}Co_{0.2}O₂ cells. The use of higher concentrations of the FRA is known to reduce the flammability of the electrolyte solution, thus, a concentration range was investigated (i.e., 5 to 20 percent by volume). The desired concentration of the FRA is the highest amount tolerable without adversely affecting the performance in terms of reversibility, ability to operate over a wide temperature range, and the discharge rate capability.

The use of fluorinated carbonates, much in the same manner as the incor-

poration of fluorinated ester-based solvents, was employed to reduce the inherent flammability of mixtures. Thus, electrolyte formulations that embody both approaches are anticipated to have much lower flammability, resulting in enhanced safety.

This work was done by Marshall C. Smart and Ratnakumar V. Bugga of Caltech and G. K. Surya Prakash and Frederick C. Krause of the University of Southern California 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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