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Materials and Coatings Development: From Bench Scale to Application

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ABSTRACT

Materials research, and more specifically the development of novel coating methods, has been a central focus of Eltron R&D since the company's inception over 25 years ago. Eltron has developed, synthesized and characterized materials ranging from nanoparticles to thin films and ceramics for a wide range of applications in the aerospace, semiconductor, energy and automotive industries. In addition to traditional materials development methodologies, Eltron is exploring revolutionary approaches that include catalytic-microwave assisted processes, novel approaches to composite molding and chemical vapor deposition. This paper covers several of the materials and coatings developed for a wide range of applications. A general overview of each technology will be provided which will include highlights of the technology, a status report on the current state of the technology and potential applications.

Keywords: coatings development, scale-up, plating of aluminum, ionic liquids, ceramic refractory coating, carbon nanotubes

Introduction

Eltron Research & Development was founded in 1982 and currently occupies a modern 25,000 square foot research and development facility in Boulder Colorado. The company has 20 years' experience in the production of ceramics, metals, glasses, composites and cermets (ceramic/metal composites) as well as dense and porous coatings. A significant portion of their intellectual property is related to materials research, including polymers, multifunctional composites, ceramics, novel paints and coatings, chemical energy storage systems, materials molding, nanotubes with coatings and new coatings. In this paper we have attempted to provide a short summary of three material coating-related technologies developed at Eltron in order to familiarize the reader with the scope of our materials research.

Aluminum electroplating via ionic liquid

Environmentally-friendly plating processes that impart corrosion resistance are in very high demand and needed to replace processes that use highly toxic materials, such as cadmium or hexavalent chromium and volatile organic compounds (VOCs). Aluminum electroplating provides excellent corrosion protection and can be used in applications where components are exposed to high temperatures. Aluminum is an ideal coating choice due to its low toxicity, excellent corrosion resistance and price. The majority of aluminum electroplating is currently performed from aluminum-containing organic baths or from high temperature molten salts. However, the process that currently dominates the market involves electroplating at 100°C with pyrophoric alkyl-aluminum compounds and toluene, a highly flammable organic solvent. With the current process, there is a need for deposition of dense, non-porous aluminum films on hardware components without alkylaluminum precursors and volatile organic co-solvents.

Eltron Research has developed a room temperature ionic liquid process for the application of protective, highly dense aluminum coatings (Fig. 1). Our electrodeposition technology enables high-quality aluminum electroplating within a benign, room temperature ionic liquid, eliminating the use of highly reactive and dangerous precurors and organic co-solvents - the first step toward reducing the environmental impact of current competing processes. The technology represents development of the first commercial, affordable, ambient-temperature molten salt plating process. Commercial applications include protection of hardened steel aerospace components such as landing gear, fasteners, and engine mounts, as well as aluminum alloy

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aerospace components such as electrical connectors. The United States Navy has expressed interest in this technology based on the proof-of-concept that Eltron has provided.

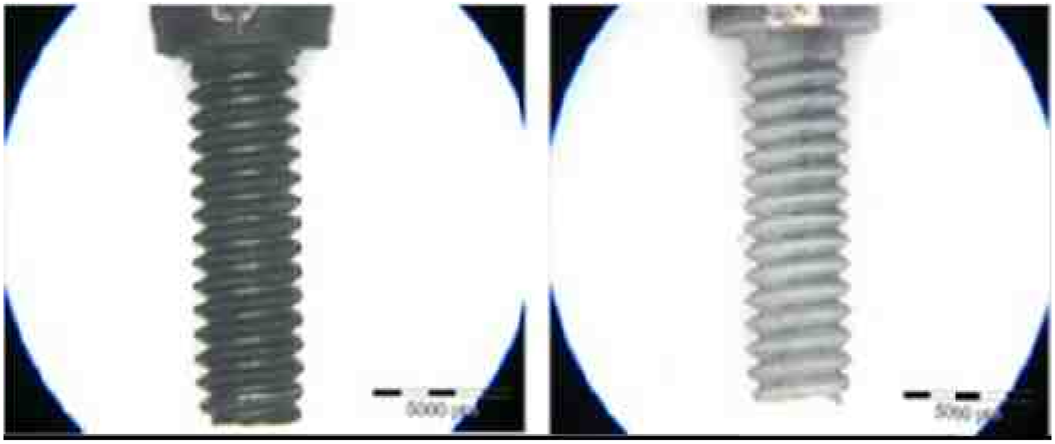

Figure 1 - Left: Uncoated bolt; Right: Same bolt coated using Eltron's room temperature ionic liquid process.

Ceramic refractory coating

Pulsed power applications, including high power microwave tubes, coaxial plasma guns and electron beam tubes, generate large electric fields which increase the energy of flashover events. In a plasma gun, insulators must be able to withstand the high voltage pulse power conditions as well as the high temperature plasma created. Refractory coatings on high voltage plastic insulators have demonstrated advantages created by combining the low weight, low cost, resilient electrical insulation of plastic insulators with the ruggedness of ceramics. Significant effort has been expended in geometrically designing insulators to minimize the electric field at the triple junction in order to maximize surface flashover voltage. As the shape of the insulator becomes more complex, the use of plastic becomes more desirable. For low cost and high performance, light weight refractory ceramic-coated plastic insulators are needed to enhance high temperature and plasma resistance of insulators for pulse power systems. Organic coatings are valuable for many applications. However, they provide poor refractory protection and often produce dangerous VOCs. Ceramic insulators offer many benefits, however they are typically heavy, difficult to manufacture and easily crack.

Eltron has developed refractory paint that has excellent electrical, thermal and mechanical properties. This commercially-available flexible ceramic paint creates a tough refractory layer. It cures at room temperature, is water-based, and contains no volatile organic compounds (VOCs), organic binders or plasticizers. As seen in Fig. 2, the paint shows excellent adhesion and flexibility even when applied to a flexible substrate. The paint can be applied by spraying, dipping or brushing. Surface preparation methods have been developed for Teflon®, chloropel, Kapton®, Mylar®, butyl-coated Nomex®, high-density polyethylene and other materials such as glass, quartz, fiberglass and alumina and various metals. Originally developed for high voltage plastic insulators used by the U.S. Air Force in pulsed power applications, our refractory paint coatings can be applied to plastic insulators of complex shape and large size with excellent adhesion (Fig. 3).

Tests for surface flashover and electrical discharge resistance show that plastic insulators coated with the refractory paint possess superior insulating and breakdown properties, when compared to solid ceramic insulators, while retaining the weight advantages and mechanical resilience of plastic insulators. Due to its inorganic nature and the lack of organic binders or plasticizers, the refractory paint displays excellent thermal properties in high temperature and plasma conditions. It can withstand thermal cycling between -196°C and 200°C on a variety of substrates. Test results also illustrate the paint's low flammability. Insulation coated with the refractory paint resists ignition, shrinkage and melting (Fig. 4). A refractory paint-coated square and uncoated square of high density polyethylene were exposed to the direct flame of a propane torch for 20 seconds at a distance of 2.5 cm. In addition to considerable structural damage and shrinkage, the uncoated plastic sample ignited and continued to burn for one minute after the torch was removed. The coated plastic self-extinguished in only three seconds.

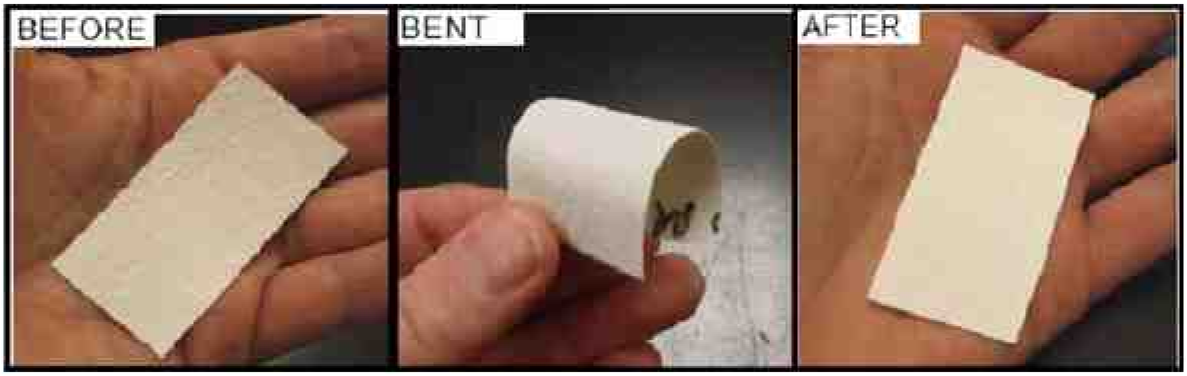

Figure 2 - Photographs of ceramic coated (Mylar®) flexible substrate showing no cracking or delamination during 180º bending.

Figure 3 - Complex plastic shape coated with refractory paint.

Figure 4 - Refractory paint coated square (left) and uncoated square (right) of high density polyethylene after exposure to direct flame of a propane torch for 20 seconds, at a distance of 2.5 cm (1 in.).

Carbon nanotubes for advanced materials

Applying carbon nanotubes (CNTs) as reinforcement in structural composites has been widely pursued, since the strength of CNTs was first calculated to be far greater than that of steel, with a density only one-sixth that of steel. The calculations showed that SWNTs are the strongest materials yet discovered, which would ordinarily make them an excellent choice for use in reinforcing polymeric composites. However, this has not been found to be the case. The primary difficulties encountered with incorporating carbon nanotubes into polymeric materials (both thermosets and thermoplastics) has been the incompatibility of their graphene surface with the hydrocarbon surfaces of the polymeric matrices and the insolubility of the CNTs themselves. The obvious differences in material surface properties of the polymers from those of CNTs yielded characteristics that do not facilitate good adherence between the materials.

Eltron researchers found a way to overcome the incompatibility issues and developed polymers of unprecedented strength and toughness via composites of epoxy and single-walled carbon nanotubes (SWNTs). The issues of insolubility were overcome by using the polymer poly(m-phenylenevinylene-co-2, 5-dioctoxy-p-phenylenevinylene) (PmPV) to function as an interfacial bridge between the SWNTs and the material in which the tubes are being dispersed. Furthermore, the tendency of the polymer backbone in PmPV to adopt a helical configuration acts to promote the winding of the polymer around both individual SWNTs and multiple SWNT ropes. The interaction between the PmPV and SWNTs is purely mechanical, so there is no incursion into the bond structures of the SWNTs. Attaching various functional groups to the side chains of the PmPV polymer makes covalent bonding possible between the composite reinforcement (the PmPV/SWNT) and the final composite's matrix (Fig. 5). The result is a strong, lightweight, non-corrosive, solvent-resistant material with desirable mechanical, electrical and thermal properties. With only 1% loading, the epoxy strength can be increased by 45% or more and the toughness can be increased by 100% or more. In addition, our SWNT composites do not suffer from the same delamination issues that plague other carbon fiber composites.

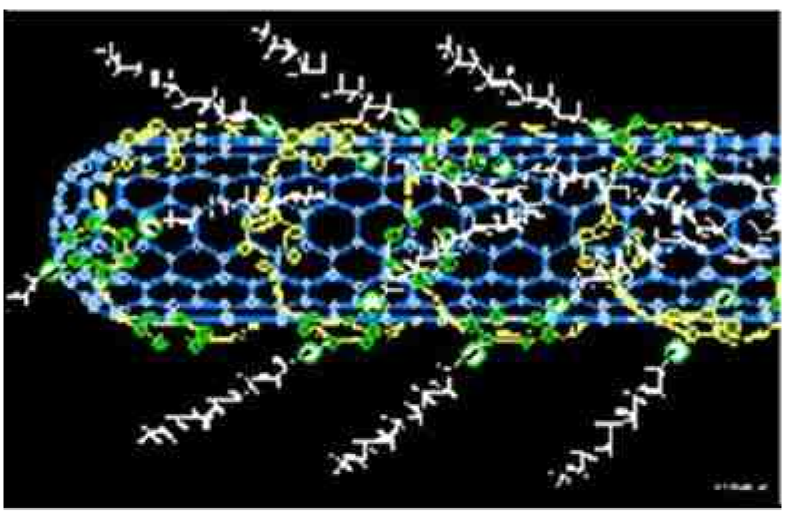

Figure 5 - SWNT with the polymer PmPV wrapped around its outer surface. The side chains protruding from the nanotube surface are being modified to bind chemically with the epoxy precursors during curing of the composite.

Today, Eltron researchers have successfully developed PmPV-coated, single-walled carbon nanotubes, achieved 1% loading in epoxy resulting in 45% increase in strength and a 100% increase in toughness, and identified means by which to functionalize the side chains for greater dispersion, which should result in a significant, further improvement in strength.

Summary

As noted earlier, Eltron's contributions to the area of materials R&D, and to coatings in particular, are much more diverse than the three technologies detailed here, including advanced separation membrane studies, specifically our hydrogen separation membrane technology. Other key projects have included the development of materials and a novel system for oxygen separation via thermal swing adsorption, catalytic coatings to remove soot from gasoline engines, highly stable membranes for nanofiltration and reverse osmosis, conductive polyimide films for spacecraft, regenerable sulfur adsorbents, and thermally stable, high temperature ceramic coatings for ferrous materials.

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About the authors

Dr. Alonso Lozano-Morales is an Electrochemical Engineer at Eltron Research and Development, Inc. His current responsibilities include development and scale-up of Eltron's advanced membrane system for H_2 separation and CO_2 capture. Prior to joining Eltron R&D, he was a Principal Scientist at Faraday Technology, Inc. where he developed electrochemical engineering processes that enabled the deposition and/or removal of materials (metals, super alloys and ceramics), so as to be utilized in the manufacturing of products. He received his B.S. from Universidad de Sonora, Mexico and his Ph.D. degree from Louisiana State University, Baton Rouge, LA. All the above degrees are from the Department of Chemical Engineering.

Dr. Erick Schutte currently serves a dual role at Eltron as both a Senior Chemist II and the company's SBIR Program Coordinator. Dr. Schutte has a diverse research background having run or participated in numerous SBIR Phase I and Phase II materials and catalysis projects during the past nine years. Past research has included environmentally-friendly magnesium and magnesium alloy anodization, corrosion-resistant coatings for hot pipe protection, radiation-resistant polyimide composites, materials and systems design for temperature swing oxygen separation, regenerable sulfur and ethylene adsorbents, and development of catalysts for use in chemical looping systems. Dr. Schutte's SBIR role allows him to bridge the gap between the R&D and the marketing of Eltron's technologies as he works closely with Eltron's business development department to develop commercialization strategies and intellectual property.