AMPTIAC
ADVANCED MATERIALS AND PROCESSES TECHNOLOGY
The AMPTIAC Newsletter, Fall, 1999
Volume 3, Number 3

Thermal Spray Coatings Replace Hard Chrome Plating on Aircraft Components

Guest Editorial
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Washington, DC

Functional hard chrome plating is a technique that has been in commercial production for over 50 years and which is a critical process associated with manufacturing and maintenance operations on aircraft, vehicles, and ships. However, current and anticipated environmental regulations, together with concerns related to performance, are leading to the qualification of other types of coatings as replacements for chrome. Within the Department of Defense (DoD), hard chrome plating is widely used, with the total value of plating operations exceeding $100 million annually.

In functional hard chrome plating, chromium deposits with thicknesses ranging from 2.5 to 500 µm (0.1 to 20 mils) are obtained from passing an electric current through a solution of chromic acid and a catalyst. It is used to provide resistance to wear, abrasion, heat and corrosion as well as to provide low friction, and to restore dimensions on worn, undersized, or ground parts, where the chrome is over-deposited and machined back to the correct dimension. Vickers hardness values (300 g load) for chrome plating are generally in the range of 8.5 to 10.5 GPa. Hardness starts to decrease above 220˚C; consequently, chrome plating should not be used for wear resistance in applications where the service temperature exceeds 400˚C.

Hard chrome plating utilizes chromium in the hexavalent state (hex-Cr), which is a known carcinogen. As a result, the Environmental Protection Agency (EPA) has issued fairly stringent air emission standards for hex-Cr under the Clean Air Act Amendments of 1990, and the Occupational Safety and Health Administration (OSHA) has established permissible exposure limits (PEL) for hex-Cr in the workplace at a level of 0.1 mg/m^3. However, recent studies have indicated that there is a significantly increased cancer risk at this level and that there is still an increased cancer risk at an exposure level one order of magnitude lower. OSHA is expected to issue a proposed new regulation in the Fall of 1999 and preliminary indications are that the new data will support lowering the PEL to a range of 0.005 to 0.0005 mg/m^3. Based on a study conducted by the Naval Sea Systems

continues, page 2 ➤

Spotlight on Technology: Phthalonitrile-Based Materials Exhibit Improved Properties for High Temperature Applications

Phthalonitrile polymers, under development at the Naval Research Laboratory (NRL), are considered to be a significant breakthrough in the development of resin based composites for high temperature aerospace, missile, marine, and commercial platforms. This article discusses the temperature and processing capabilities of phthalonitrile resins as well as ongoing developmental efforts concerning the processing, testing and component fabrication of phthalonitrile-based composite materials. A brief overview of the opportunities offered by this class of materials is presented with sufficient details to be of interest to those considering the selection of advanced materials for high temperature systems.

Resin: Phthalonitriles are a class of high temperature thermoset resins that cure by monomeric addition reactions. It has been shown that a variety of chemical additives can be used to control polymerization rates, which is a very desirable feature for composite or component fabrication. Phthalonitrile resin products exhibit excellent oxidative and thermal stability and show no signs of a glass transition at temperatures at least 150˚C (~ 300˚F) higher than state-of-the art high temperature poly-

continues, page 12 ➤

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HVOF Thermal Spray Coatings

continued from page 1

Command in 1996, the total annual additional cost to the Navy for compliance with the lower PEL would be $46 million for all operations utilizing hex-Cr. Since hard chrome plating is one of the major activities that utilize hex-Cr, it is clear that the increased cost will be in excess of $10 million annually. The chrome plating industry has acknowledged that if a PEL of less than 0.010 mg/m^3 is implemented, many plating operations will have to shut down.

Particularly in the military and civilian aerospace sector, there are concerns related not only to the increased cost and liability associated with more stringent OSHA and EPA regulations, but also to the in-service performance of chrome plating. In the past, chrome has been the best available coating to meet industry and military requirements. Increased material performance that would lead to reduced life-cycle costs is becoming more essential due to the fact that aircraft are being flown considerably longer than originally anticipated and because of the steadily decreasing funding for aircraft maintenance within the DoD. In addition, the increased thrust-to-weight requirements on gas turbine engines require materials with improved performance characteristics. Therefore, there is a strong incentive towards identifying new coating technologies that not only eliminate the environmental and health problems associated with hard chrome plating, but also demonstrate increased performance compared to chrome. The technology area that is most promising in terms of meeting these goals, and which also is sufficiently developed for demonstration/validation and technology insertion, is thermal spraying.

Previous technology assessments (e.g., Northwestern University, sponsored by the Defense Advanced Research Projects Agency) have concluded that high-velocity oxygen-fuel (HVOF) thermal spraying is the best available technology for hard-chrome replacement on components used in aircraft, ground vehicles, and machinery. Because of the size of HVOF spray guns and the fact that it is a line-of-sight process, there are many components currently being chrome plated that are not amenable to HVOF spraying, such as internal diameters of cylinders less than 25 cm (10 inches) in diameter. However, for some applications such as aircraft components, it has been determined that up to 75% of all parts currently being chrome plated could be replaced by parts sprayed with HVOF coatings.

HVOF thermal spray systems have been commercially available for more than 10 years. An HVOF thermal spray gun utilizes an internal combustion jet to generate supersonic gas velocities of approximately 1800 meters/second. Combustion fuels that are mixed with oxygen in the gun include propylene, acetylene, propane, hydrogen, and kerosene, with combustion ignition and gas control being fairly straightforward. The types of materials that can be deposited include metal alloys, ceramic/metal composites, and polymers; powders of the coating materials are injected directly into the combustion region of the gun under automatic control. The semi-molten powder particles are accelerated in the high-velocity gas stream exiting the nozzle of the gun to velocities of approximately 400 meters per second and impact on the surface of the part being coated. Generally, the standoff distance between the gun and surface of the material being coated is between 15 and 30 cm. For most HVOF coatings, the porosity is less than 1%, the oxide content is less than 1%, and the bond strength usually exceeds 80 MPa (10,000 psi). These properties make HVOF the most attractive of the thermal spray techniques for chrome replacement. The instantaneous deposition rate is approximately 50 µm (0.002 inches) per minute although in normal operation the HVOF gun is mounted on a robot or other articulating arm and the component being coated is also manipulated. It generally takes less than 20 minutes to deposit a 100-µm-thick coating onto a 0.5-meter-long, 0.1-meter-diameter cylinder. Through judicious use of air jets on

The AMPTIAC Newsletter is published quarterly by the Advanced Materials and Processes Information Analysis Center (AMPTIAC). AMPTIAC is operated by IIT Research Institute, a not-for-profit contract research organization, and is a DoD-sponsored Information Analysis Center, administratively managed by the Defense Information Systems Agency (DISA), Defense Technical Information Center (DTIC). The AMPTIAC Newsletter is distributed to over 20,000 materials professionals around the world.

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the component during deposition, surface temperatures can be kept below 175˚C (350˚F); therefore, many temperature-sensitive materials can be coated. Figure 1 is a photograph showing deposition of an HVOF tungsten carbide/cobalt (WC/Co) coating onto a Boeing 737 nose landing gear cylinder.

Figure 1: HVOF thermal spraying of WC/Co onto a nose landing gear cylinder from a Boeing 737. The cylinder is approximately 0.6 m long.

In the early 1990's there were several studies conducted that compared the properties of different HVOF coatings to hard chrome. These generally showed superior results for HVOF, but did not lead to wholesale technology insertion efforts. In 1996, the DoD Environmental Security Technology Certification Program (ESTCP) awarded a project to demonstrate and validate HVOF coatings as a viable replacement for hard chrome on aircraft components. As a result, a tri-service/industry group, the Hard Chrome Alternatives Team (HCAT), was created to execute the program. The HCAT effort includes all aspects required for complete technology insertion. One of the key objectives is to show that the performance of HVOF coatings is superior, not just equivalent, to hard chrome.

The initial HCAT tasks involved conducting fatigue, corrosion, and wear tests on coupons in order to determine the effects of the coating process on substrate materials, not to qualify the coatings for specific applications. Three substrate materials, 4340 steel, 7075 aluminum alloy, and PH13-8Mo stainless steel were selected as representative of materials onto which hard chrome is currently applied. Two HVOF coatings were selected for evaluation in comparison to hard chrome—WC/Co (83%/17%) and Tribaloy 400, a cobalt-molybdenum-chromium alloy.

It is well known that application of hard chrome plating causes a fatigue debit in most materials, especially high-strength steels. Therefore, it was the objective of the fatigue study to deposit the HVOF coatings such that the fatigue debit would be minimized. Three types of test specimens were utilized: (1) 0.63-cm (0.25-inch) diameter smooth round bar, (2) 0.63-cm (0.25-inch) diameter hourglass bar, and (3) 1.40-cm (0.55-inch) x 0.51-cm (0.20-inch) rectangular Kb bar. For the smooth bar, the length of the gage area was 1.9 cm. All specimen fabrication and testing was conducted by Metcut Research, Inc. with the exception of the testing of the hourglass bars, which was conducted by the Air Force Research Laboratory. The 4340 steel was heat-treated to 1.8-1.9 GPa (260-280 ksi) to maximize sensitivity to fatigue effects. Both low-cycle fatigue (LCF) and high-cycle fatigue (HCF) tests were conducted and S/N curves generated over a wide range of maximum load conditions. The LCF tests were conducted in axial strain control using a 2 hertz triangular waveform which was switched to load control at 9 hertz for the duration of the test if failure did not occur in the first 24 hours (172,800 cycles). The HCF tests were conducted in load control using a 60 hertz sinusoidal waveform. For the LCF tests, the R ratio (Pmin/Pmax) was 1.0; for the HCF tests, R was 0.025. Figure 2 shows the S/N curves for the 4340 smooth round bar specimens, including uncoated 4340. The data for the hard-chrome-coated specimens indicated a substantial fatigue debit, as expected. However, the data for both of the HVOF coatings were within the statistical uncertainty of that for the uncoated specimens, indicating essentially no loss of fatigue strength. Data for the hourglass and Kb bar specimens were similar to that for the smooth bar specimens.

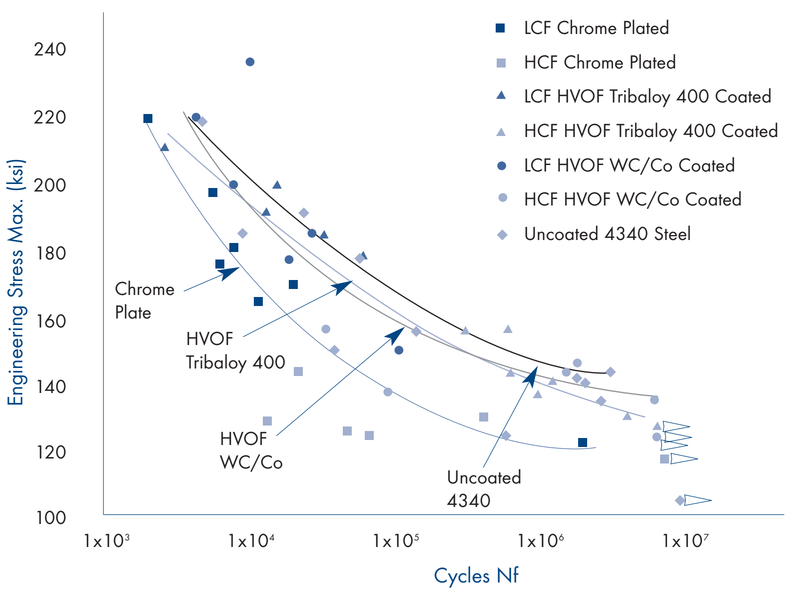


Figure 2: S/N fatigue data for smooth bar uncoated (baseline) 4340 steel specimens and 4340 specimens coated with hard chrome, HVOF WC/Co (83%/17%) and HVOF Tribaloy 400.

Three types of corrosion studies were conducted on flat plate specimens fabricated from the three base materials. The first was the ASTM B117 salt fog test and the second the GM9540P/B cyclic corrosion test, both of which were conducted in a Q-Fog Model cabinet with the appropriate test protocol stored in the controller memory. The third test was atmospheric exposure at the Navy Marine Corrosion Test Facility at Key West, Florida. All of these tests indicated that the performance of the HVOF coatings exceeded or were equivalent to hard chrome plating.

All of the above studies demonstrated

continues, page 7 ➤

University of Delaware Teams with Industry in Intelligent Manufacturing Program for Composites

Resin transfer molding (RTM) is a process used to fabricate automotive, aircraft, and ship structures from fiber-reinforced polymer-matrix composites. While RTM has been in existence for more than 50 years, it has seen a dramatic increase in both research and applications over the past decade. This is due in large part to the high potential for cost savings through near-net-shape manufacturing of complex parts to exact tolerances. However, further expansion of this technology will occur only if the process itself can be made truly cost-competitive.

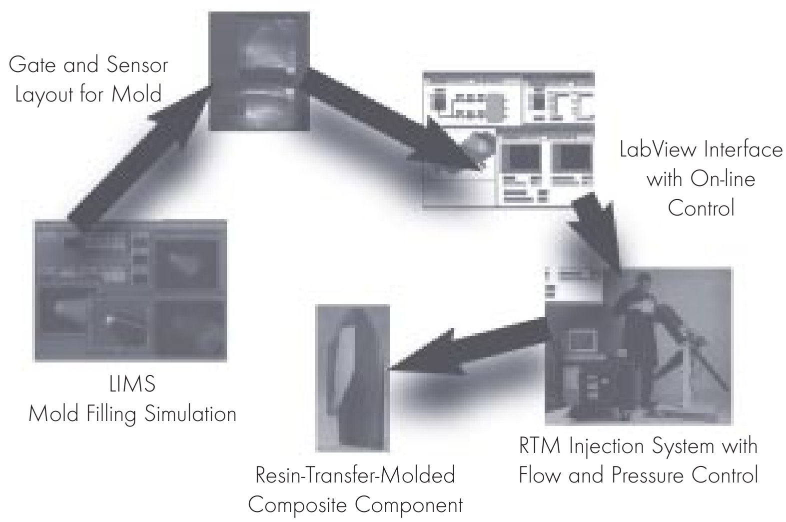
"We know that RTM is a feasible process for near-net-shape manufacturing, but we have to bring science to the manufacturing process if it is to become economical," says Dr. Karl V. Steiner, Executive Director of the University of Delaware Center for Composite Materials. "With materials comprising only a fraction of total product cost, we have to take a harder look at the process itself in terms of cost-competitiveness."

To that end, Steiner and Prof. Suresh Advani of UD's Department of Mechanical Engineering are leading a program aimed at applying a manufacturing science approach to the automation of RTM. Funded by the Office of Naval Research (ONR), the Advanced Materials Intelligent Processing Center (AMIPC) links UD researchers with industry and government to address critical issues relevant to both civilian and defense applications. Boeing is involved as a major manufacturer of aircraft components and Honeywell's role is as a controls specialist. A fourth partner, IITRI, recently co-organized with UD a workshop in Annapolis, Md., to facilitate technology transfer with other major players in the field of on-line sensing and control for RTM.

"A major cost factor in RTM," says UD's Prof. Advani, "is production of the mold, which includes inlet gates and exit vents for resin injection, as well as sensors to detect resin flow and cure." Advani is credited with developing LIMS (Liquid Injection Molding Simulation), a comprehensive simulation code for mold filling. The code is being modified and integrated into the AMIPC program to explore a variety of filling scenarios and enable investigation of the influence of various processing conditions. These include temperature, pressure, vent and gate locations, and sensor positions. Based on this cost-effective and rapid off-line simulation, the researchers are able to identify the optimum locations for gates, vents, and sensors. "This approach leads to clear cost avoidance in tool preparation and modification of existing tools," says Advani.

Boeing's participation in the program is enabling application of the research to the production of actual parts. The company is evaluating RTM as a potential technology for military and commercial aircraft applications. Phase I of the AMIPC has focused on optimizing the mold filling parameters during RTM. Honeywell has been contributing to the development of on-line control software for this process step. The on-line control system is based on a LabVIEW platform and provides real-time feedback from the process and the mold to the user on the shop floor. Several control strategies are implemented to track process conditions, detect anomalies, and provide solutions to compensate for any deviations, thus reducing the scrap rate and the need for costly repairs. Under Phase II, these control strategies are being combined with injection hardware from Radius Engineering, which features flow and pressure control for intelligent RTM, towards pre-commercial units, two of which will be installed at the University of Delaware and Boeing.

"The U.S. Navy is seriously considering composites—with their inherent advantages for signature management, weight, and maintenance cost reduction—for use on structural components for its current aircraft and ships as well as for its next-generation aircraft and surface ship and submersible combatants," says James J. Kelly, Program Officer of ONR's Materials Division. "A fundamental understanding of the materials science combined with detailed knowledge of the manufacturing environment is the key to successful implementation of composites processing. The AMIPC team is addressing these needs of national importance through its unique approach to manufacturing science."



For more information contact Dr. Karl V. Steiner, Center for Composite Materials, University of Delaware, Newark, DE 19716-3144, phone (312)831-2310, fax (302)831-8525, email: steiner@ccm.udel.edu ■

Publicize Your Program

This issue of the AMPTIAC Newsletter features a Spotlight on Technology article about promising new phthalonitrile materials. This article was submitted to AMPTIAC by scientists at the Naval Research Laboratory. Previous editions of the newsletter have featured articles from U.S. Government organizations such as the Air Force Research Laboratory, NIST, and from the Department of Energy's Continuous Fiber Ceramic Composite program. Private contractors such as Pratt & Whitney, as well as members of academic institutions such as the University of Delaware, Clarkson University and SUNY at Stony Brook have also contributed articles.

The publication of articles raises the visibility of research and technology often resulting in increased or renewed interest in a particular technology. AMPTIAC has received many questions and comments from its readers. In fact, AMPTIAC is still getting comments and questions regarding a recent article on the Titanium Matrix Composite Turbine Engine Component Consortium (TMCTECC) published nearly a year ago. The TMCTECC article is also featured on the Aeronautics Learning Laboratory for Science, Technology, and Research (ALLSTAR) (http://allstar.fiu.edu). David Brumbaugh's article on polyaniline coatings for metals has resulted in several government and commercial in-situ trials.

AMPTIAC has a mailing list of over 20,000 individuals in the advanced materials community, including persons working in government agencies, for defense contractors, as well as research and academic institutions. All Newsletters are available from the AMPTIAC web site (http://amptiac.iitri.org), increasing the newsletter's circulation. AMPTIAC's web analysis has consistently indicated the newsletter portion of the web site is the most visited area of the entire web site.

AMPTIAC invites members of the advanced materials community, particularly those members involved in U.S. Government sponsored research, to submit articles of interest to the newsletter. Articles can be on virtually any topic concerning advanced materials, including research, new product innovation, technology transfer, or new applications for relatively mature technologies. The AMPTIAC Newsletter has earned a reputation as a quality publication, thanks to its previous contributors. Still, we have only scratched the surface of what the newsletter can be. If you would like to submit an article for publication or, if you have an idea for an article or a series of articles, please contact David Rose, AMPTIAC Director, or Barbara Severin, Newsletter Editor. ■

AMPTIAC Wants Your Contributions

We hope you find this issue of the AMPTIAC Newsletter useful and interesting. You can help us to better serve you by your contributions, such as:

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- Your suggestions for AMPTIAC data products and services
- Technical articles, opinion pieces, tutorials, news releases or letters to the Editor for publication in the Newsletter

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Your contributions are always welcome.

New Chief Scientist Announced

Air Force Research Laboratory Selects Dr. Kenneth Harwell

Wright-Patterson AFB, OH – The Air Force Research Laboratory recently announced its new chief scientist is Dr. Kenneth E. Harwell of the University of Alabama at Huntsville.

"We've conducted a methodical search for someone with the right mix of technical prowess, leadership ability, hands-on experience, and stature within the research community," said Maj. Gen. Richard R. Paul, AFRL commander. "Dr. Ken Harwell meets all those criteria. We're delighted to have him join the AFRL team."

The laboratory plans, manages and conducts research and development activities for the Air Force to advance the entire range of aerospace and interrelated technologies. It has an annual budget of $2.5 billion, a government work force of over 6500 people, and is located at 10 major sites throughout the United States.

"We're thrilled Dr. Harwell is coming on board," added Dr. Don Daniel, AFRL executive director. "We sought an exceptionally qualified leader for this technical position, and we're confident he's the right person for AFRL."

"I'm very pleased and very excited to be joining the excellent research team of the new Air Force Research Laboratory," Dr. Harwell said. The laboratory was created in October 1997 from the consolidation of all Air Force science and technology assets.

"I'm looking forward to working with General Paul, Dr. Daniel, and the AFRL technology directors and their staffs to provide the leadership for performing the research needed to keep the nation at the forefront of aerospace technology throughout the next century," Dr. Harwell said. "I'm awed at the magnitude of the task ahead of us as we continue to make the Air Force research program the best in the world."

Dr. Harwell will be the most senior technical advisor to the lab's commander. He will assist the commander in managing the technical content of AFRL's scientific and technology portfolio, while maintaining a university position through the Intergovernmental Personnel Act. Currently, he is the senior vice president for research and associate provost at the University of Alabama, positions he has held for almost 10 years.

The AFRL chief scientist evaluates the lab's total technical program, identifies gaps and analyzes advancements in a variety of technical fields to determine their influence on lab programs and objectives. The person in this position also fosters collaborative efforts with foreign countries, other services, universities and industry. In addition, the chief scientist represents the lab to other DoD laboratories, major aerospace companies, NASA, FAA and international research organizations.

As chair of the AFRL Research Council, the chief scientist provides executive leadership to the chief scientists of the lab's technology directorates to ensure the highest professional standards of technical quality are maintained. He also evaluates prospective candidates for critical positions and recommends people for senior-level technical positions.

The lab's work force of military and civilian personnel is a diverse mix of professional scientists, engineers, administrators and technicians. They work in a highly specialized, geographically separate complex of laboratory, office and support facilities.

The laboratory has research sites at Wright-Patterson AFB, OH; Kirtland AFB, NM; Eglin AFB, FL; Tyndall AFB, FL.; Bolling AFB, DC; Hanscom AFB, MA; Edwards AFB, CA; Brooks AFB, TX; and in Rome, NY and Mesa, AZ, as well as offices in Europe and Japan. ■

continued from page 3

what might be called the technological viability of HVOF coatings as a replacement for hard chrome plating, but full-scale production validation requires a significant amount of additional work. The key to full qualification of HVOF coatings is the involvement of all appropriate stakeholders which, within the military aerospace sector, means repair depot technical representatives and engineering authorities, weapons systems program managers or single item managers, and structural engineers from the three services and from the original equipment manufacturers (OEMs). Because hard chrome plating is used on a variety of aircraft components, this means that there will be different stakeholders involved in the qualification process for each type of aircraft component. As a result, the HCAT has joined with two other DoD organizations, the Joint Group on Pollution Prevention (JGPP) and the Propulsion Environmental Working Group (PEWG), to execute separate projects to fully qualify HVOF coatings on landing gear, hydraulic actuators, propeller hubs, helicopter dynamic rotor components, and gas turbine engine components. The execution plan for each project first involves the development of Joint Test Protocols (JTP) which define all of the necessary testing required for qualification. These generally include coupon testing such as fatigue, corrosion, and wear of the selected HVOF coatings on base materials appropriate for that type of aircraft component, and actual component testing which involves installing coated components into test rigs and into operational aircraft where performance is tracked over extended periods of time. In addition to preparation and execution of the JTP's, the projects also involve establishing production-level HVOF facilities at the appropriate military aircraft maintenance facilities, conducting training of personnel, conducting cost analyses, and developing standards and specifications for application, grinding, and stripping of the HVOF coatings on the categories of aircraft components.

At present, the most developed of the projects is the one on landing gear which is a joint project between the U.S. and Canada. In 1997 the Canadian Department of National Defence (DND) and Industry Canada (IC) became interested in the HCAT program because of the considerable number of military aircraft in DND and because Canadian companies manufacture more than 2/3 of the landing gear used on military and commercial aircraft in North America. As a result, a partnership was formed between the U.S. and Canadian teams which led to the execution of a formal Project Arrangement between the two countries. The preparation of the Joint Test Protocol for landing gear is complete and calls for extensive coupon and component testing by the U.S. team on HVOF WC/Co and by the Canadian team on HVOF WC/Co-Cr (86%/10%-4%). Fatigue, corrosion, and wear coupon testing and component evaluations have been initiated. For the latter, an F/A-18E/F main landing gear with several HVOF-coated components is being tested in a rig at Boeing St. Louis, and HVOF-coated landing gear components are being evaluated on Navy P-3 and E-6A aircraft currently in service, with anticipated additional flight testing to be done by the Air Force on the F-15 and F-16, and by Canada on the C-130.

There have been several cost and time studies conducted on the replacement of chrome plating with HVOF on landing gear. One study was conducted at the Jacksonville Naval Aviation Depot, where approximately 20,000 parts are chrome plated annually. Results indicated that more than 14,000 of the parts could be coated with HVOF and that with full production implementation of the technology, the depot could save several million dollars over a 15-year time period. Figure 3 shows the results of a labor-hour and time study done by Sulzer Metco for the processing of Boeing 737 nose landing gear pistons. The bars in the chart indicate the number of either labor hours or process hours for each step in the production processes. It is apparent that significant savings in process hours can be realized by converting to HVOF, principally due to a reduction in coating deposition time and the elimination of the lengthy post-deposition bake-out required with chrome.

At the completion of the entire program, it is expected that HVOF thermal spray coatings will be in production in many military manufacturing and maintenance activities. The increased performance of HVOF-coated components should ultimately lead to reduced maintenance, thus lowering the total-cost-of-ownership to DoD for its aircraft. The commercial aircraft industry is also moving independently to embrace this technology with several airlines such as Delta and Lufthansa conducting flight evaluations of HVOF-coated landing gear components. Overall, this is an example of where concern over environmental regulations are helping drive organizations toward the adoption of a superior technology, with the ultimate result being reduced costs and a cleaner environment. ■

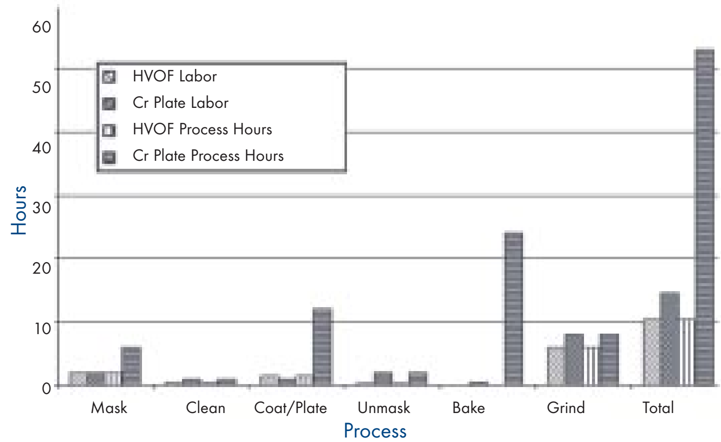

Figure 3: Labor and process hours associated with the various steps in repairing a Boeing 737 nose landing gear inner cylinder piston using chrome plating and HVOF WC/Co thermal spray coating (courtesy Bruce Bodger, Sulzer Metco)

NRL Develops Advanced Aerogels

Scientists in the Naval Research Laboratory's (NRL's) Chemistry and Optical Sciences Divisions have developed composite aerogels for use as high-surface area electrodes, catalysts and battery structures, advanced thermoelectric materials, and as architectures around which to design chemical, physical, and optical sensors. Since aerogels provide both high surface areas and highly open spaces, they are especially well-suited to catalytic and sensing applications, where rapid transport of reactants and large, accessible surface areas are critical to performance. NRL has received external support for this program from the Office of Naval Research and the Defense Advanced Research Projects Agency.

Aerogels are an advanced class of materials composed of approximately 10-nanometer particles connected in a highly porous, three-dimensional network. Aerogels are a combination of both particles and pores, which is the key to their unique properties. The most widely studied aerogel composition is silica.

During the past year, the NRL team, led by Drs. Debra Rolison and Celia Merzbacher, has provided design flexibility and expanded the range of aerogel properties by using the gel's building block, colloidal silica sol, as a "nanoglue" to trap suspended particles or colloids into the network of the wet gel. This technique has been used to make composite aerogels of silica with a range of physically and chemically diverse particulates.

When the second phase of the composite is present above a certain threshold, its transport characteristics are imparted to the composite aerogel, even though it retains the low density and openness characteristic of pure silica aerogels. Although a carbon-silica composite aerogel is approximately 80% open space, it blocks transmission of He-Ne laser light, even though a pure silica aerogel transmits the beam of laser light with minimal scattering.

Real-world surfaces are actually nanoscale domains that differ from the underlying bulk and that dictate many of the technologically most relevant catalytic and electrical properties of the material. "Our research program on aerogels demonstrates how a fundamental effort to design and characterize nanostructured platforms leads to improvements in the properties of technologically relevant materials and the design of new materials," comments Dr. Rolison. ■

AMPTIAC Announces New Products

A Practical Guide to Statistical Analysis of Material Property

This report has been specially prepared with the materials professional in mind. It bridges the gap between the science of theoretical statistics and the hands-on world of the practicing technician. The first of its kind, this report presents important statistical analysis methods from the standpoint of material property data, demonstrating the importance and relevance of statistics in the day-to-day activities of materials engineers and designers. (186 pages)

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Life Prediction and Performance Assurance of Structural Materials in Corrosive Environments

Life prediction of structural components is vitally important to safe and cost effective operation of any system in which the materials are susceptible to environmental degradation. Performance assurance which is closely related to life prediction, is equally important to ensure that the system will operate as per design for the duration of its life. This report presents a panoramic view of this field by highlighting the variety of current approaches, identifying the limitations, and discussing directions for future efforts. (120 pages)

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Corrosion Predictive Modeling for Aging Aircraft - Critical Review & Technology Assessment

Budgetary constraints prevent acquiring new aircraft while encouraging life extension of existing aircraft far beyond the design lives. This critical review and technology assessment highlights the significant and innovative aspects of the U.S. Force program to develop a predictive model for corrosion prevention and maintenance in complex structures such as joints. This program is a major step forward in the rather complex task of modeling corrosion and predicting the life of corrodible structures with any engineering relevance. The principles that have been described in this report to generate predictive capability are generic and applicable to a variety of components and structures. (20 pages)

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Web Link: http://www.acers.org/meet/cocoa2000/default.

24th Annual Conference on Composites, Materials and Structures
Jan 24 – Jan 28, 2000
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23rd Annual Meeting of the Adhesion Society
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International Congress & Exhibition on Defense, Test, Evaluation and Acquisition
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Sparks Fly as Novel Metallic Glass Throws Off Fireworks

Berkeley, CA — Many materials can heat up somewhat when they are bent or broken, but few throw off showers of sparks as hot as those emitted when a new kind of metallic glass is shattered. For the first time, a team of researchers in the Materials Sciences Division of Lawrence Berkeley Laboratory has measured the extremely high temperature of particles ejected when this unusual amorphous metal is fractured.

The alloy of zirconium, beryllium, titanium, copper, and nickel is one of the first metallic glasses that can be made in bulk and formed into strong, hard, useful objects. It was discovered by William L. Johnson and Atakan Peker of the California Institute of Technology.

Working with the discoverers to investigate the mechanical properties of the novel material, which as yet are poorly understood, a team of researchers in Berkeley Lab's Materials Sciences Division led by Robert Ritchie mounted notched specimens of the zirconium-based metallic glass in a pendulum impact device. They were startled to find that, when fractured in air, the alloy shot out showers of bright, hot sparks.

"We were measuring how much energy it took to fracture the material when we stumbled onto the light emission," says Christopher Gilbert, a postdoctoral fellow in Ritchie's group. "As it turned out, scientists at Oak Ridge had seen this last year – but we've managed to measure the associated temperatures and to explain the mechanism for the first time."

In air, when struck by the pendulum weight, the notched specimens snapped and sent out bright sparks whose color corresponded to a blackbody temperature of 3,175 degrees Kelvin. The same experiment in a nitrogen atmosphere produced no visible sparks, but emission was detected in the infrared at 1,400 K.

"So-called fracto-emission is familiar in brittle insulating solids and somewhat less familiar in ordinary metals," says Gilbert, "but emissions of this intensity are unprecedented in ductile polycrystalline metals. And so far as we know, fracto-emission has never been quantified in amorphous metals."

Digital camera images, plus the experiments in pure nitrogen, showed that the sparks in air were caused by burning particles thrown off from the fracture surface. When the broken specimens were examined under a scanning electron microscope, blobs of melted material were seen on the fracture surface. The heat generated in breaking the metallic glass was enough, apparently, to ignite freshly exposed metal particles.

"Zirconium and titanium will burn in air, if you get them hot enough," says Ritchie, "but the real question is the temperature we observe in nitrogen –1,400 K in the absence of intense oxidation and pyrophoric activity."

Gilbert says, "When the metallic glass is broken, the deformation is highly localized in narrow bands, which generates intense heat from plastic work" – rather the way a wire gets hot if bent back and forth rapidly. Melting observed on the fracture surfaces means local temperatures must have exceeded 935 K, the temperature at which the metallic glass liquifies.

Team member Joel Ager surmises that the temperature rises rapidly as the material is deformed "partly because metallic glass has terrible conductivity for a metal. It can't get rid of the heat. But this can't be the whole story."

Commercial Use

Unlike pure metals and most metal alloys, metallic glasses have no regular crystalline structure. This lack of long range order or microstructure is related to such desirable features as strength and low damping – the ability of some of these alloys to deliver a really big bounce – which is one reason why the premier use for zirconium-based metallic glass is in the manufacture of expensive golf club heads.

Only recently has it been possible to obtain metallic glass in enough bulk to make a golf club head or to perform extensive mechanical testing. In the past, to prevent segregation and crystallization of the melt required such rapid cooling – in only about a thousandth of a second, at a rate of about a million degrees Celsius per second – that only very thin wires and ribbons could be formed.

Zirconium-based metallic glasses can be cast in bulk because they can be cooled much more slowly, at about 10 degrees C per second; they achieve their glassy, disordered state by alloying metals with dramatically different atomic sizes and chemical characteristics. The alloy studied by Ritchie's group, for example, is two-fifths zirconium and one-fifth beryllium.

William Johnson of Caltech and his colleagues at Amorphous Technologies International of Laguna Niguel, California, pioneered the development of these alloys and their commercial uses. Golf clubs made of zirconium-based metallic glass have unusual springiness, a "soft" feel, and an almost ideal density between that of stainless steel and titanium (currently the connoisseur's choice); they also demonstrate, however, that even slow-cooling metallic glass doesn't cool slowly enough for really large castings. For the time being, at least one of the dimensions must be under four inches.

Nevertheless, the properties of bulk metallic glasses – their high strength-to-weight ratios, high hardness, excellent wear properties, good forming and shaping qualities, and their unusual magnetic properties as well – hold promise for many important applications. Ritchie's group is pursuing a wide range of measurements, including electrochemical studies by team member and graduate student Valeska Schroeder.

"We're interested in determining which properties of these new materials can be attributed to their individual constituents and which to the amorphous nature of metallic glass," says Ritchie. "Valeska has already shown that the notion that metal corrosion in the zirconium-based glass would be alleviated because of its amorphous nature is simply not true. The crystalline and glassy microstructures show very similar pitting potentials, in a variety of chemical solutions."

As for unexpected light emissions from the zirconium-beryllium alloy, Ritchie says, "These extremely high temperatures aren't Polywater, not a delusion – they're real. And they demand explanation."

The researchers reported their unusual findings in the June 21, 1999, issue of Applied Physics Letters.

The Berkeley Lab is a U.S. Department of Energy national laboratory located in Berkeley, California. It conducts unclassified scientific research and is managed by the University of California. ■

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Properties of Intermetallic Alloys, Volume II, Silicides (1994)

This volume contains evaluated and analyzed data on the thermophysical and mechanical properties of silicides of cobalt, iron, molybdenum, niobium, nickel, rhenium, tantalum, titanium and zirconium. (570 pages)

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NASP Bibliographic Database (1998)

This searchable database contains bibliographic records for nearly 600 technical reports written by contractors and government personnel supporting the National Aerospace Plane (NASP) program. These reports address various materials development efforts and technologies including high temperature intermetallics, ceramic matrix composites, and carbon-carbon. Also included are references to oxidation-resistant coatings for these high temperature materials. Access to the database is Windows-based and user friendly.

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imides (Tg = 310˚C). Because polymerization is an addition type reaction with no volatile components being formed during curing, the final product is uniform and shows minimal void formation. As a result, composite materials can be fabricated by short term processing either through a conventional autoclave lay-up or by casting or automated procedures such as resin infusion (RIM) or resin transfer molding (RTM), and filament winding (FW). The capacity to regulate polymerization rates makes it possible to achieve resin cure flows that are appropriate for the fabrication of thick and complicated composite structures.

Relative to other organic polymers, the mechanical characteristics of phthalonitrile resins are comparable or superior. Table 1 compares the mechanical properties of phthalonitrile with PMR-15 (high temperature imide class) and both a general and high performance epoxy resin.

Composites: The exceptional high temperature stability and favorable processibility of phthalonitrile polymers have motivated the establishment of several new efforts in the development of phthalonitrile based composite materials. Current work includes optimization of resin processing procedures, fabrication and characterization of composites with various reinforcement materials, and generation of material property databases in the high temperature regimes (300˚C and higher).

In conjunction with The Naval Air Warfare Center (NAWC) in Patuxent River, Maryland, NRL, has initiated studies to streamline composite processing by shortening resin cure times. There is also interest in investigating alternate curing techniques such as microwave and electron beam methods. Tests of mechanical property retention after exposures up to ~600˚C (1100˚F) have been initiated for both the long term and temperature spiking conditions that are associated with missile and aerospace applications. Preliminary tests show excellent results.

At this time both glass and carbon fiber reinforced panels have been fabricated using traditional lay-up and autoclaving techniques. The mechanical properties determined for these panels are found to be comparable or superior to those of similar polyimide (i.e. PMR-15) based composites. Moreover, phthalonitrile offers the advantage of cast processing. In the case of polyimides, for example, labor intensive bagging and vacuum conditions are required to remove solvents (added to maintain flow during lay-ups) and volatiles (formed during the curing stage). Failure to remove these solvents and volatiles results in void formation as they vaporize during the high temperature cure and postcure stages. For phthalonitriles, flow conditions are maintained by controlling polymerization rates through the adjustment of curing additive quantities and temperature. Table 2 shows a comparison of the room temperature mechanical property data for PMR-15 and phthalonitrile composites.

Recently, GKN Westland Aerospace, Inc. reported a rather extensive high temperature database for resin transfer molded phthalonitrile composites fabricated with woven graphite reinforcement.[6] GKN is interested in a cost effective substitute for titanium parts in aircraft engines. Data was collected for simulated operational conditions including high humidity and temperature. Essentially, these data show that phthalonitrile composites retain their mechanical properties over a very broad temperature range, -54˚C to 343˚C (-65˚F to 650˚F) unlike composites based on BMI's (bismaleimide) resins. The room temperature properties of the GKN fabricated phthalonitrile composite are as follows: tensile strength of 90 ksi; compression after impact of 22 ksi; and interlaminar shear

Table 2. Mechanical Properties of Unidirectional Composite Panels [5]
(* denotes values measured for a panel made from different prepreg batches using phthalonitrile sized fibers)

Property	Phthalonitrile/IM7	PMR-15/IM7
0˚ Tension		
Strength, MPa	2000 [2400]* ± 20	2500 ± 175
Modulus, GPa	183 [162]* ± 3.7	146 ± 4.4
Strain, %	1.0 [1.36]* ± 0.07	1.6 ± 0.064
90˚ Tension		
Strength, MPa	41 ± 4.1	29 ± 2.03
Modulus, GPa	10 ± 0.80	9 ± 0.27
Strain, %	0.4 ± 0.048	0.5 ± 0.15
0˚ Flexure		
Strength, MPa	2350 ± 47	1530 ± 76.5
Modulus, GPa	174 ± 3.5	122 ± 4.9
Strain, %	1.3 ± 0.23	1.3 ± 0.078
90˚ Flexure		
Strength, MPa	80 ± 1.0	-
Modulus, GPa	11 ± 0.99	-
Strain, %	0.6 ± 0.024	-
Short Beam Shear		
Strength, MPa	85 ± 1.7	105 ± 2.1

Table 1. Neat Resin Properties

Property	Phthalonitrile [1]	Imide PMR-15 [2-3]	Epoxy* [4]	Cyclo-aliphatic Epoxy Cure [4]
Tensile Strength (MPa)	80 ± 5.6	43-84	41-59	89
Tensile Modulus (GPa)	4.4 ± 0.22	4	2.7-4.1	6.3
Elongation at break (%)	1.2 ± 0.06	1.4-2.5	1.1-4.9	2.1
Flexural strength (MPa)	80 ± 7.2	76	86-116	159
Flexural Modulus (GPa)	4.2 ± 0.13	3.2	2.7-3.9	6.4
Fracture Toughness, Gic(J/m^2)	120-130	87		
Glass transition temp (˚C)	>450	327		
Water Uptake (% in 24 h)	0.2	0.4		
(% in 2 weeks)	0.6	1.6		

*cast samples of bisphenol-A, cycloaliphatic and tetrafunctional epoxies having cyclic diamine cures

Spotlight on Technology

continued from page 1

strength about 60 ksi. Additional testing showed virtually no changes in the room temperature shear strength of composites immersed in nine solvents likely to be encountered during aircraft service (i.e. methyl ethyl ketone, acetone, trichloro ethane 1,1,1 (MIL-T-81533), isopropyl alcohol, propylene glycol, aircraft lubricating fuel and lubricating oil, and salt water) for up to thirty days. Solvent sensitivity is generally indicated by a breakdown in composite shear strength.

Properties such as low weight, reduced maintenance, corrosion resistance, and low magnetic and acoustic signatures have interested the marine community in organic matrix composites for surface ship and submarine components and structures. Despite these benefits, however, strict flammability codes restrict the use of organic materials on sailing vessels particularly in a submarine environment. Fire hazard assessment is made through the determination of the heat released by chemical reactions occurring in a given weight or volume of material in a fire and by the time to ignition. Heat release is an indicator of damage that could be incurred during combustion of that material. Heat release standards for submarines are described in MIL-STD 2031. Collaborative efforts involving NRL and the Naval Surface Warfare Center (NSWC) at Carderock, Maryland have shown that the phthalonitrile composite meets these standards and in fact exhibits significantly lower heat generation than many other polymer composites. Table 3 shows the heat release data for glass reinforced phthalonitrile composites as compared to the standard and to similar organic matrix composites.

Gl/phthalonitrile composites also show a highly favorable ignition temperatures (Tig) as compared to other resins. For example, the time to ignition for Gl/phthalonitrile is about 30% longer than for the corresponding bismaleimide or epoxy systems. Table 4 shows ignition times for Gl/phthalonitrile compared to five other polymer composite systems. Extensive toxicity tests have not been done. However, in general glass-reinforced pthalonitrile produces lower quantities of CO (40ppm), HCN (trace), and HCI (non-detectable) as compared to glass-reinforced vinyl ester, epoxy, bismaleimide, phenolic, and polyimide composites.

Component Manufacture, Scale-up and Commercialization: At this time, the phthalonitrile resin system is sufficiently mature for prototype parts construction, scale-up, and testing for potential applications. The phthalonitrile monomer is easily converted to a prepolymer version (B-stage) which can be stored at room temperature indefinitely for later usage. While the cost of research quantities of the monomer is currently an area of concern, monomer price should be significantly reduced as production batches reach commercial size. Additionally, because the major portion of composite component cost is found in processing and manufacture (85% relative to 15% for the resin and installation) low-cost processing such as RTM or FW (filament winding) are expected to keep production costs competitive with those of the present polymer based composites.

Table 4. Time to Ignition for Polymeric Composites [7]

	Heat Flux in kW/m²			
	25	50	75	100
Time to Ignition in Seconds				
MIL-STD 2031	300	150	90	60
Composite				
Gl/phthalonitrile	NI*	NI	84	59
Gl/vinyl ester	180	67	34	24
Gl/epoxy	535	105	60	40
Gl/bismaleimide	503	141	60	38
Gl/phenolic	NI	214	57	25
Gl/polyimide	NI	175	75	55

*No Ignition

Research efforts at NRL, NAWC, and several industrial facilities are currently optimizing RTM methodologies for the manufacture of components that can be used in the temperature range of -54˚C to 343˚C (-65 to 650˚F). This work involves investigations of short term cure effects on material property retention. Baseline comparisons will be made against the present commercial high temperature polyimide systems, Avimid-N and PMR-15.

Attracted by potential cost savings from automated processing capabilities, the Air Force has instituted an SBIR program focused on RTM process optimization for the fabrication of aircraft engine components. Results of this work are also expected to have a significant impact on the DoD Joint Strike Fighter (JSF) Program.

NRL and NSWC-Carderock have initiated collaborative research and development efforts to explore possible phthalonitrile transitions as fire resistant materials in the Navy fleet. This work includes trade-off assessments of potential composite applications internal to the submarine pressure hull as well as identification of cost effective fire sensitive composite applications that are most promising for future implementation. In conjunction with these efforts there are investigations to determine the technical feasibility and suitability of vacuum-assisted resin transfer mold processing (VARTM) of phthalonitrile composites and phthalonitrile-vinyl ester laminates for shipboard use. VARTM is now the Navy preferred procedure for the manufacture of high quality polymer composite parts. Phthalonitrile veneers would be used to provide fire resistance to vinyl ester composites that are presently favored for use on surface ships.

Within its Firesafe Composite Program (*Proceedings-43rd Annual SAMPE Conference*, October 1998) Electric Boat Corporation is now evaluating phthalonitrile composites for a variety of military and domestic platforms requiring ma-

continued ➤

Table 3. Heat Release Values for GI/polymer Composites Compared to MIL-STD 2031 [7]*

MIL-STD and Composite Type	Peak heat release rate (kW/m²) at a radiant heat flux of 75 kW/m²	Peak heat release rate (kW/m²) at a radiant heat flux of 100 kW/m²
MIL-STD 2031	100	150
Gl/phthalonitrile	98	106
Gl/bismaleimide	245	285
Gl/polyimide	78	85
Gl/vinyl ester	165	187
Gl/phenolic	97	133
Gl/epoxy	217	232

Gl = glass woven fiber

Recent US Patents

Patent Number	Title
5,940,733	Method Of Making Polysilicon/Tungsten Silicide Multilayer Composite On An Integrated Circuit Structure
5,940,675	T222 Production By Powder Metallurgy
5,940,246	Disc Drive Hydro Bearing Lubricant With Electrically Conductive, Non-Metallic Additive
5,939,514	Coating Composition Having Extended Storage Stability And Method For Producing Wear Resistant Coatings Therefrom
5,939,335	Method For Reducing Stress In The Metallization Of An Integrated Circuit
5,939,216	Fiber Reinforced Ceramic Matrix Composite And Method Of Manufacturing The Same
5,939,213	Titanium Matrix Composite Laminate
5,939,184	Polyolefin-Based Composite Material Containing A Stratiform Silicate And Production Process Therefor
5,939,147	Scandia, Yttria-Stabilized Zirconia For Ultra-High Temperature Thermal Barrier Coatings
5,939,146	Method For Thermal Spraying Of Nanocrystalline Coatings And Materials For The Same
5,939,144	Method And Composition For Diffusion Treatment Of Ceramic Materials
5,939,041	Process And Apparatus For Removing Metal Oxide Particles In The Regeneration Of Pickling Acids
5,939,013	Process And Apparatus For The Production Of High Strength Polymer Composite Structures
5,939,011	Method For Producing A Mandrel For Use In Hot Isostatic Pressed Powder Metallurgy Rapid Tool Making
5,939,007	Method For Manufacture Of A Fiber Reinforced Composite Spar For Rotary Wing Aircraft
5,939,006	Method For Forming A Composite Airfoil Structure
5,938,998	Method And Apparatus For Manufacturing Composite Insulators
5,938,934	Dendrimer-Based Nanoscopic Sponges And Metal Composites
5,938,862	Fatigue-Resistant Lead-Free Alloy
5,937,708	Ceramic-Metal Composite Assembly
5,937,321	Method For Manufacturing Ceramic Multilayer Circuit
5,936,861	Apparatus And Process For Producing Fiber Reinforced Composite Objects
5,936,718	Method For Detecting Fiber Misalignment In Composite Manufacturing
5,936,025	Ceramic Polymer Composite Dielectric Material
5,935,891	High-Loading Adsorbent/Organic Matrix Composites
5,935,887	Ceramic Filter And Method For Preparing Same
5,935,722	Laminated Composite Of Magnetic Alloy Powder And Ceramic Powder And Process For Making Same
5,935,713	Protective Resistant Composite Material And Projectile Resistant Composite Body Formed Therefrom ■

terials with improved, maintenance, low weight, and better corrosion performance. Actual sized phthalonitrile composite components have been fabricated and are ready for potential military and commercial testing.

Summary:

Based on their exceptional thermo-oxidative stability, excellent processing capabilities, mechanical property retention, and fire and chemical resistance, phthalonitrile polymers represent a major step forward in the development of organic matrix composites for high service temperature performance. They are now considered sufficiently mature for process optimization, configuration, scale-up, and testing to meet the requirements of specific applications.

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Spotlight on Technology continued from page 13

SACMA Releases Industry Statistics

Washington, DC – The Suppliers of Advanced Composite Materials Association (SACMA) recently released statistics on worldwide shipments of carbon fiber composite materials for 1998. During this period, carbon fiber shipments totaled 23,041,073 pounds, valued at $580,200,000. The table below gives totals for 1992 through 1998.

The SACMA statistics are drawn from actual member company data and are estimated to represent 85 percent of the shipments of carbon fiber for advanced composite applications within North America, Western Europe, and the Far East, during the period reported.

Worldwide Advanced Composite Carbon Fiber Shipments for 1992-1998

YEAR	POUNDS	U.S. DOLLARS
1992	13,002,812	$374,100,000
1993	14,598,544	$384,900,000
1994	17,425,452	$461,400,000
1995	19,714,671	$464,800,000
1996	20,672,741	$489,240,000
1997	25,964,530	$621,410,000
1998	23,041,073	$580,200,000

■

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The AMPTIAC technical inquiry service is offered free of charge for the first eight hours of service. AMPTIAC will use all available resources, including Ph.D. level staff members, to ensure that our support is adequate to address your needs. Requests that may require additional time are charged to reflect the amount of effort and level of expertise required to provide a useful answer. Under no circumstance will a user be charged for services without a prior agreement to do so.

AMPTIAC's inquiry service could help save time and money. For more information, contact AMPTIAC by any of the means listed on the back cover of this newsletter.

AMPTIAC

ADVANCED MATERIALS AND PROCESSES TECHNOLOGY

Inside this Issue ...

Thermal Spray Coatings Replace Hard Chrome Plating on Aircraft Components

Spotlight on Technology: Phthalonitrile-Based Materials Exhibit Improved Properties for High Temperature Applications

And more ...

Please, if you wish to contact us you may do so at…

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AMPTIAC is a DoD Information Analysis Center Administered by the Defense Information Systems Agency, Defense Technical Information Center and Operated by IIT Research Institute

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