

In The News

Recent Conferences

TSS and NRC Host Successful Sensors and Controls 2004 Meeting

ASM International's Thermal Spray Society (ASM-TSS) held a two-day workshop meeting "Sensors and Controls 2004" on Oct 26-27, 2004. The meeting was hosted by the National Research Council Canada's (NRC-CNRC) Industrial Materials Institute in Boucherville, near Montréal, Québec, Canada, and was cosponsored by ASM-TSS and Tecnar Automation Ltd.

Organized locally by a team comprising Christian Moreau (NRC), Mike Gevelber (Boston University), Jean Gabriel Legoux (NRC), and Luc Pouliot (Tecnar Automation Ltd), the meeting featured 22 presentations over the two days, with a keynote address from 2004 ASM President and former TSS President Dr. Robert C. Tucker Jr., FASM, a panel discussion featuring seven of the speakers and six tabletop exhibits. A total of 117 attendees from nine countries attended the meeting where presenters from industry and academia showcased the current needs and expectations of large OEMs, day-to-day challenges faced by job shops, implementation of on-line controls, plasma plume diagnostic techniques, equipment manufacturer's visions for the future, and advanced diagnostics and controls.

Visits during the meeting included tours of the facilities at Tecnar Automation Ltd and a tour of the thermal spray facilities at NRC. Tecnar Automation Ltd also spon-

sored cocktails and the dinner at the Hotel Mortagne in Boucherville. Copies of the presentations from this meeting will be available from ASM International.



Andrew Nicoll



Robert Tucker



News from Institutes and Research Centers around the World

EMPA—Materials Science and Technology History

EMPA was founded in 1880 as a federal institute for testing building materials with close links to the Swiss Federal Polytechnic Institute (ETH Zurich). It was established in the wake of a severe rail accident that killed about 80 passengers, the result of the collapse of a steel bridge 5 km from Basel. To investigate whether the accident was caused by poor quality steel or by incorrect design was one of the first tasks undertaken by the young EMPA. In the following decades, many more areas of expertise were estab-

lished in addition to steel and concrete, such as road engineering, wood, lightweight materials, high-performance ceramics, polymers, combustion engines, fuel, lacquer and paintings, corrosion, fibers for textiles, biomaterials, and so forth. Besides traditional material testing, research activities became more and more dominant, with the result that in 1988 the term "Research" was added to the institute's name.

EMPA has become a leading institution for applied research in many fields, both nationally and internationally, as attested by regular peer reviews. Research areas

are nowadays focused on "advanced materials and surfaces," "materials and systems for civil engineering," "protection and well-being of the human body," "information, reliability, and simulation technology," "mobility, energy, and environment," and "nanotechnology" as cross-linked topics.

EMPA celebrates its 125th anniversary this year. EMPA belongs to the ETH domain together with other institutions like the Federal Institutes of Technology in Zurich (ETHZ) and Lausanne (EPFL), the Paul Scherrer Institute (PSI), the Swiss Federal Institute for Environmental Science and Technology (EAWAG), and

the Swiss Federal Institute for Forest Snow and Landscape Research (WSL).

The newest EMPA site at Thun was established in Jan 1994 following the transfer of a part of the material testing laboratories of the Swiss Military Department to EMPA. In total EMPA has approximately 770 employees working at three different sites. About 500 people are employed at the main site in Duebendorf, a further 220 or so in St. Gall (close to Lake Constance), and approximately 50 people at the third branch dealing with material technology and nanotechnology, located in Thun (Fig. 1).

The “Materials Technology” Section

The “Materials Technology” section of EMPA in Thun (<http://www.empathun.ch>) is subdivided into three groups:

- Thermal Spraying & Non-oxide Nanopowders
- Materials Characterization and Measurement Research
- Metal Matrix Composites (MMCs)

Main Activities of the “Thermal Spraying and Non-oxide Nanopowders” Group

The EMPA Thun is active in the research, development, processing, and characterization of metallic, ceramic, and polymer based powders and coatings. Various spraying techniques such as conventional flame spraying (FS), atmospheric plasma spraying (APS), or vacuum plasma spraying (VPS) are available using industrial-scale units. Modern equipment and in-flight particle diagnostics for measuring particle temperatures, speed, diameter, and jet position ensure the monitoring of all relevant process parameters and forward parameter development.

Basic research and applied coating developments are carried out in close collaboration with various academic and industrial partners and customers within Switzerland, Europe, and around the world. Activities range from basic research on coating formation, microstructure, and adhesion, to feasibility studies



Fig. 1 The thermal spray laboratory (left) and office and service/characterization building (right) at the EMPA Thun site

and the manufacturing of first prototypes. The strong support of the “materials characterization group” shortens the time from the first spraying tests to optimized coating results.

For comprehensive coating characterization, the following methods are available on-site: microstructure characterization, chemical analysis, mechanical testing, in situ micro- and nanomechanical testing in the high-resolution scanning electron microscope, nondestructive testing, and tribological examinations as well as various wear and corrosion tests performed according to the various national and international standards.

Access is available to a comprehensive range of information on materials, processes, suppliers, published literature, and existing standards, in the group’s own databases.

Recently, an inductively coupled plasma pilot plant was built up to produce nonoxide nanopowders such as nitrides, carbides, and even pure nanometallic powders. Thus, depending on requirements, powders with different morphologies and chemical compositions can be produced, if necessary even in large quantities.

Basic research areas include:

- Development of coating materials (metals, ceramics, and polymers, nanophased and nanostructured alloys, graded materials, and composites, etc.)
- Optimization of process parameters for different applications concerning coating density, corrosion resistance, thermal conductivity, wear resistance, electrochemical or biological behavior, and so forth
- Theoretical investigations of material/plasma interaction as well as the study of nanoparticle formation
- Feasibility studies, new applications (e.g., in the fields of fuel cells, coatings for hydropower applications, quasicrystalline coatings, nonwetting/nonstick coatings, nanostructured coatings, etc.), and technology transfer
- Basic research, for example, on adhesion/cohesion, coating microstructure, fracture, internal stresses, and so forth.

Education/Events. EMPA regularly organizes workshops and conferences on a range of topics (for further information see: www.empa.ch EMPA Akademie). In addition, EMPA further supports research for doctoral theses and the exchange of its own scientists with other institutes, as well as offering positions to post doctorates and guest researchers.

Resources

The thermal spraying laboratory at EMPA Thun is equipped with modern in-

dustrial-scale spraying units for carrying out coating development according to the requirements of industrial customers and scientific partners. The VPS process enables the deposition of functional coatings of high-melting-point or reactive materials under inert or reactive gas atmospheres. Together with a spin-off company and other industrial partners, access to different HVOF systems (with gas or liquid fuel) is available.

An inductively coupled radiofrequency plasma system was set up for nanopowder synthesis. This equipment can also be used for thermal spraying or gas phase deposition.

Instrumentation/equipment include:

- Vacuum plasma spraying: Medicoat MC 60 (Fig. 2), Torch F4
- Flame spray: Castodyn DS 8000, Metco 5P, Uni Spray Jet-GTV U 7100
- HVOF: Terojet (in collaboration)
- RF induction plasma: Torch: Tekna (PL 35), 35kW, 13.56 MHz
- In-flight particle diagnostics: Tecnar DPV/CPS 2000
- Powder laboratory equipped for powder production, sieving, mixing, grinding, heat treating, and measuring of grain-size distribution and morphology, etc.

Instruments for Characterization and Control. In order to achieve the desired coating quality in a straightforward manner and to guarantee reproducibility, modern in-flight particle diagnostics (measuring particle temperature, speed, diameter, and plume location) and continuous monitoring of all process parameters can be applied. The following characterization methods are available for investigating the quality of the sprayed coatings:

- Metallographic laboratory for microstructure characterization (optical or high-resolution scanning electron microscope, AFMs, etc.)



Fig. 2 Computer-controlled vacuum plasma spray equipment with in-flight particle diagnostics and four-fold powder feeder

- Chemical analysis (EDX, XRD, GDOS)
- Mechanical testing: for example, tensile adhesive strength measurements (according to EN 582, ISO 14916), micro-mechanical testing (pushout tests, four-point bending tests, and tensile tests, all in SEM)
- Nondestructive testing (topographical AFM/laser measurements, ultrasonic testing (EN 583-1), and so forth)

Some of the wear tests available according to various national and international standards include:

- ASTM G 32 (Cavitation Erosion)
- ASTM G 65 (Dry Sand/Rubber Wheel)
- ASTM G 73 (Liquid Impingement Erosion) modified
- ASTM G 75 (Slurry Abrasivity “Miller Number”)
- ASTM G 76 (Solid Particle Impingement in combination with G 73)
- ASTM G 99 (Pin-on-Disk)
- ASTM G 133 (Linearly Reciprocating Ball-on-Flat Sliding Wear)
- ASTM G 134 (Erosion of Solid Materials by a Cavitating Liquid Jet)

Corrosion tests include:

- DIN 50021 or ASTM B 117, ASTM G 85 (Salt Spray (Fog) Testing)
- ASTM B 368 (Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Test))
- ASTM G 87 (Conducting Moist SO₂ Tests)
- Locally resolving electrochemical tests, and others

Scientific Partners

EMPA is collaborating with partners from academia and industry around the world, for example (in alphabetical order) from the following countries: Canada, Czech Republic, France, Great Britain, Germany, Italy, Japan, United States, and so forth.

Selected Project Highlights

Insights into Microstructure in Relation to Coating Properties

The microstructure of thermally sprayed coatings can be varied over a wide range by adjusting spraying process parameters. The microstructure itself significantly influences the physical and mechanical properties of thermally sprayed deposits. However, reliable relationships among the spraying parameters, the microstructure of the deposits, and the resulting properties are still not fully understood. This is due to the complex defect structures with pores ranging from a few

nanometers to tens of micrometers, micro- and macrocracking, residual stresses, and the anisotropy of the coating properties. A research project is aimed at elucidating such cross correlation in a more general way by using novel analytical techniques on a wide range of microstructures of technologically relevant nickel-base alloys (Ni, NiCr, NiCrBSi, NiCrAlY). The coatings were manufactured by different thermal spray processes in industrial use such as plasma spraying (APS), vacuum plasma spraying (VPS), water-stabilized plasma spraying (WSP), flame spraying (FS), wire arc spraying (WAS), and high-velocity oxyfuel (HVOF) spraying.

The microstructural investigations have been focused on the pore and crack structure, which are considered to be the most important microstructural features of influence on the chemical and physical properties of coatings.

SANS. The use of small-angle neutron scattering (SANS) combined with standard techniques (image analysis, XRD, porosimetry, etc.) affords an enhanced level of understanding of microstructure control. SANS offers nondestructively quantitative results on the anisotropy of pores and cracks on a large volume.

SANS characterization is a quantitative method, and it allows separate characterization of void systems within the microstructure. The SANS measurements were carried out at the SINQ facility at the Paul Scherrer Institute in Switzerland. Cross sections of the coatings adhering to their metallic substrates were prepared and measured with the neutron beam entering the samples edge-on. The measurements were made with neutrons of 5 Å in wavelength and at sample-to-detector distances varying from 2 to 20 m.

Typical scattering curves illustrate that the absolute scattering intensity $I(Q)$ decreases with the fourth power of the scattering vector Q (Q^{-4} Porod Regime), which is observed over a wide range in Q . The related Porod-constants were found to differ by up to a factor of 5 for the same starting materials sprayed by different

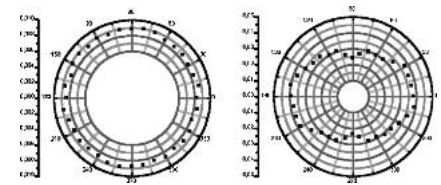
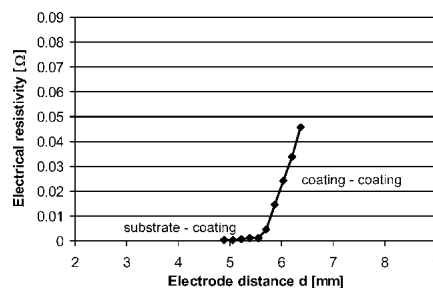


Fig. 3 Porod constants apparent surface area (m^2/m^3) as a function of the rotation angle. Isotropic behavior can be seen for the WSP-NiCr coating (left), whereas the APS-NiCr coating (right) shows pronounced anisotropy.

methods and process parameters. From the Porod constants, the specific internal surface area of the voids and the anisotropy of the pores and cracks can be determined (Fig. 3).

The corresponding coating properties were investigated by different physical methods, for example, ultrasonic testing, electrical conductivity, fracture toughness, and so forth, which were scaled down and modified within this project for application to thermally sprayed coatings.

Electrical Conductivity. A four-electrode wafer-testing device was adapted for use with thin cross sections of thermally sprayed coatings using tungsten probes in a micropositioning system. An example of typical resistivity versus electrode distance data for NiCrAlY samples both perpendicular to the substrate surface and in-plane is shown in Fig. 4. The curve (left) reflects both the conductivity of the substrate and of the coating itself. To investigate the electrical anisotropy of such coatings, the same tests were done for sectioned samples for in-plane direction (Fig. 4, right).

The two most sensitive methods for detecting coating inhomogeneities and anisotropy were found to be SANS and electrical resistivity measurements.

Electrochemical Coating Characterization. A new portable scanning electrochemical cell was developed allowing fast, nondestructive and quantitative measurements at a lateral resolution comparable to the coating thickness. Measurements on thermally sprayed nickel-base coatings performed by the scanning tech-

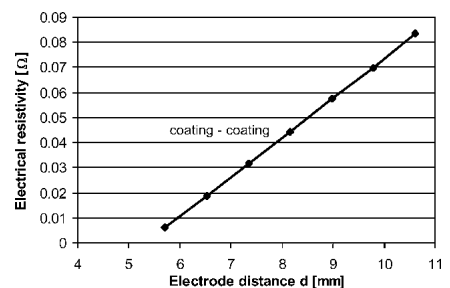


Fig. 4 Microresistivity measurements for an APS NiCrAlY coating perpendicular to the substrate surface (left) and in-plane (right)

nique showed good agreement with the results obtained later by the more qualitative and time-consuming salt-spray test used industrially.

A quantitative locally resolved characterization of potential corrosion is now possible within a few minutes, allowing fast comparisons of different coatings regardless of their surface topography and geometry. For a representative measurement, the scanning area is smaller and the acquisition time required is significantly shorter than for salt-spray testing.

Conclusions. Microstructure optimization using this new level of understanding will increase the control of the properties and reliability of materials in surface technology that are widely used in industry. This investigation was performed within a EUREKA consortium and aimed at understanding microstructure-property relationships. The fracture toughness was found to be mainly influenced by the larger defects, whereas electrical conductivity and elasticity are dominated by the large contribution to the overall internal surface from the numerous small pores.

The results indicate a significant anisotropy of the coating properties. This suggests the careful use of quantitative values of any coating property for engineering applications assuming isotropic behavior.

These investigations were partially supported by EUREKA/KTI grant agencies under project Σ! 1973 “Thermetcoat” and performed by EMPA, CH (N. Margadant, St. Siegmann), Paul Scherrer Inst., CH (T. Keller, W. Wagner), Sulzer Metco, CH (G. Barbezat), ANL, USA (J. Ilavsky), Inst. of Plasma Physics, CZ (J. Pisacka), Skoda Materials and Techn. Res., CZ (P. Fiala, R. Enzl).

Coating Adhesion and Shear Testing

A New Simplified Shear Test for the Characterization of Adhesive and/or Cohesive Coating Failure without Adhesives. Adhesive bond strength measurements of thermally sprayed layers according to given standards (e.g., ASTM C 633, EN 582, or ISO 14916) are technically limited, time intensive, and require expensive test equipment not normally available in coating shops. The biggest problems are the chemistry, the application of the adhesive, and the limited bond strength. Depending on the coating chemistry and the structure of the coating, bond strength values can be much higher than that of the adhesive. Additionally, the adhesive may penetrate the coating forming a compound, which influences the value of the bond strength of the original coating. To investigate this, a research project was initiated within the European Frame-

work “CRAFT Standards, Measurement & Testing.” The aim was to develop a new, low-cost, simplified shear test method for characterizing the mode and

failure value of thermally sprayed layers in a more reliable and less limited manner. This shear test does not need an adhesive and yields more information about

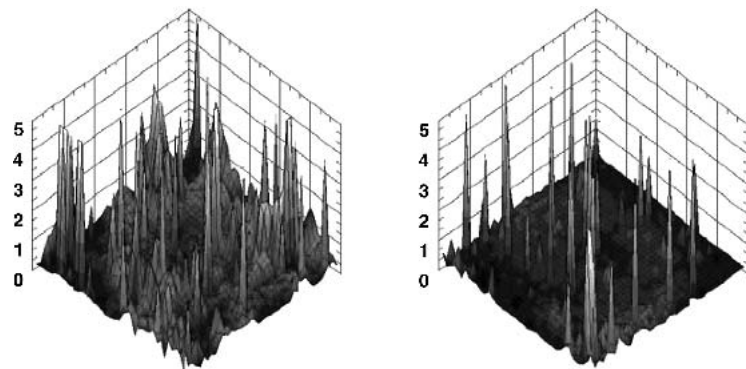


Fig. 5 Electrochemical maps ($18 \times 18 \text{ mm}^2$) of the current densities of a porous nickel flame sprayed sample (left) and a dense coating produced by VPS (right)

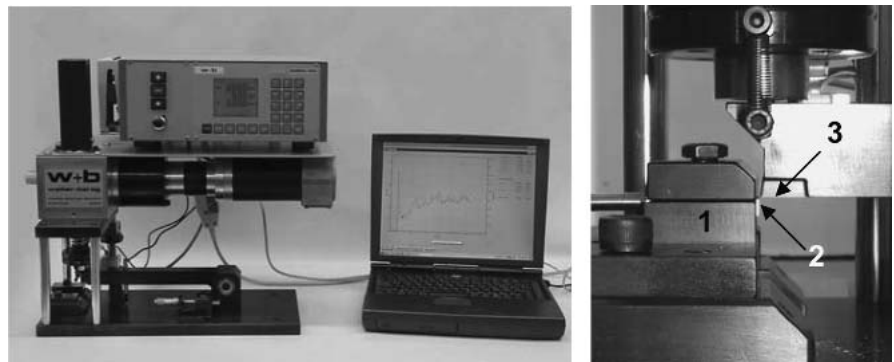


Fig. 6 Prototype of the shear test equipment (left) and close up (right) of the sample fixation (1), sticking out coating (2), and shear plate (3) viewed from the side

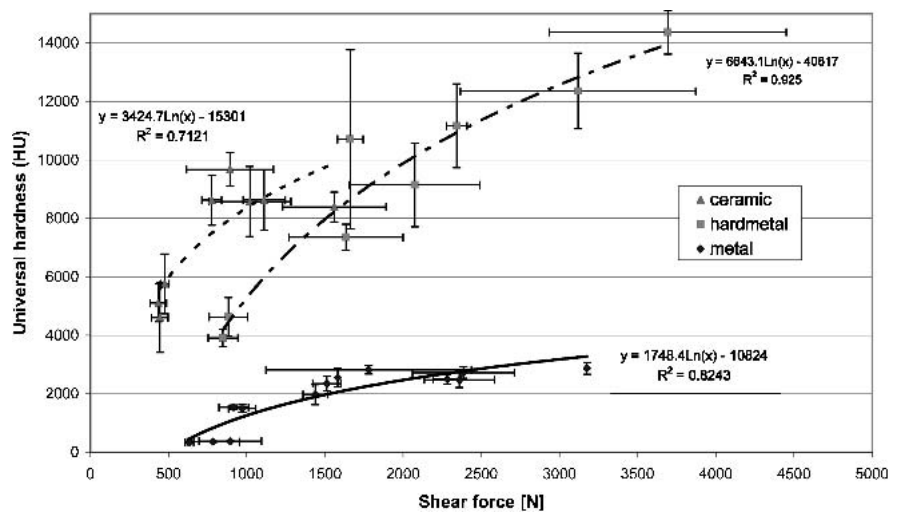


Fig. 7 Results of the shear test on metallic, ceramic, and hard metal coatings sprayed with seven different types of thermal spray systems (wire arc, wire and powder flame, atmospheric plasma, vacuum plasma, HVOF (gas and liquids), cold gas). The maximum shear force is depicted against the universal hardness (HU).

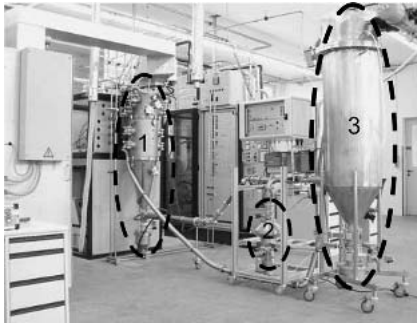


Fig. 8 Inductively coupled plasma pilot plant for nanoparticle research and production consisting of 1, RF plasmatron and synthesis chamber; 2, in situ nanopowder sampling filter; and 3, specially designed nanoparticle filtering/sampling device for large quantities

coating quality than the conventional tensile tests.

Seven different types of spraying techniques and several coating materials

(metals, hard metals, and ceramics) have been evaluated using the standardized bond strength method and the new shear test. The results were compared and correlated (Fig. 7).

Using the logarithm of the universal hardness as a weighing factor, a clear linear tendency can be seen for all the metals, hard metals, and ceramic coatings measured. This offers a new type of interpretation of adhesion and cohesion.

This project was supported by Fifth EU-Framework and BBW grants under the CRAFT “Shear Testing” project and performed by IFAM Bremen, DE (H. Grutzner), EMPA, CH (St. Siegmann, M. Dvorak), GTVmbH, DE (K. Nassenstein), Buser Oberflächentechnik AG, CH (S. Isch); Walter+Bai AG, CH (A. Walter); Metallisation Limited, GB (T. Lester); OBZ Dresel & Grasme GmbH, DE (D. Grasme); Euroflamm Italiana srl; Erling Jansen Aps (H. Møller).

Nanopowder Research

Research investigations and the production of nonoxide nanopowders were started three years ago. An inductively coupled plasma system was built up using a PL-35 torch (TEKNA) operating at power of 35 kW and frequency of 13.56 MHz. Within this thermal plasma, primarily solid, but also liquid or gaseous precursors can be vaporized and subsequently quenched out of the gas phase to retain the nanostructure. The materials synthesized to date are carbides (WC, TiC), nitrides, and pure metals (Si, Ti, Cu, etc.). Basic research on particle formation and plasma/material interactions are being carried out.

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Industrial News

New Benchmark Achieved With the Development of Nanoscale Coatings Using Electric Arc Spraying

The NanoSteel Company reports the achievement of nanoscale coatings through the twin wire arc spraying process. Analogous to achievements in the HVOF and plasma spray coatings, this new development with the SHS717 cored wire was enabled by extensive alloy design studies that revealed specific combinations of atom ratios that readily form metallic glass structures at cooling rates in the range of thermal spray (10^4 K/s to 10^5 K/s). During the devitrification process, which can occur either during spraying or during a secondary post heat treating anneal, the glass precursor readily transforms into a nanoscale composite structure. The refinement of microstructure to the nanoscale regime by this route is possible due to the uniform nucleation and extremely high nucleation frequency that occurs during crystallization at high undercoolings, resulting in limited time for grain growth before impingement between neighboring grains.

The as-sprayed and heat treated microstructures of the SHS717 electric arc coatings are shown in Fig. 1(a) and (b), respectively. The as-sprayed microstructure is characterized by a glassy matrix containing starburst-shaped boride and carbide crystallites ranging in size from 60 to 140 nm. The heat treated fully devitrified microstructures consist of a three-

phase matrix consisting of α -iron, carbide, and boride phases on a structural length scale of between 60 and 110 nm. The chief advantage of this approach toward developing nanostructured coatings is that the feedstock material, representing specific powder cuts for HVOF or plasma, or conventionally sized cored wire for electric arc is physically identical to conventional feedstock, which eliminates the spraying and handling problems normally associated with nanoscale particulate materials. The ability to develop nanoscale composite coatings while processing in air using conventional electric arc spray guns is revolutionary and results in coatings with excellent combinations of properties including high bond strength, hardness, wear resistance, and corrosion resistance.

Contact: Dr. Daniel James Branagan, Chief Technical Officer; tel: 208/552-5225, e-mail: dbranagan@nanosteelco.com; or Michael J. Breitsameter, VP of Marketing and Business Development; tel: 407/838-1427; e-mail: mbreitsameter@nanosteelco.com; Web: www.nanosteelco.com.

(Adapted from SprayTime IV, 2004)

Self-Lubricating NiCr Matrix Composites Resist High Heat

A composite high-temperature self-lubricating chrome-oxide-based material that is embedded with compound particles that function as solid lubricants has been announced by the Battelle/Great Lakes Technology Center, Cleveland, OH. Called PS300, the material is made by combining solid lubricants and ce-

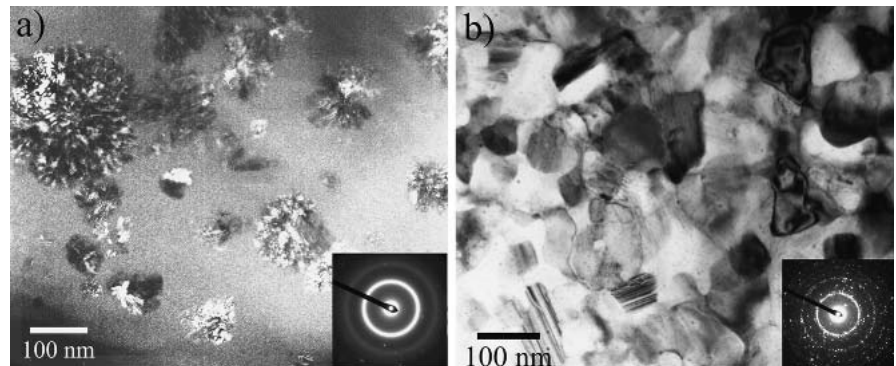


Fig. 1 Transmission electron micrographs along with their corresponding selected diffraction patterns of the SHS717 electric arc coating in the as-sprayed (a) and fully devitrified (b) (700 °C for 10 min) conditions

ramic hardeners in a nickel-chromium matrix. The material is an outgrowth of more than three decades of high-temperature tribological research at NASA Glen Research Center, also of Cleveland. It was specifically developed as a shaft coating to protect foil air bearings used in oil-free turbomachinery such as gas turbines.

PS300 reduces friction and wear in sliding contacts from below ambient to more than 650 °C (1200 °F). The material starts in the form of a powder and can be easily manufactured or applied by standard powder metallurgy methods such as thermal spraying and press-and-sinter methods.

PS304 is one particular alloy in the PS300 family. PS304 has a composition by weight of 60% NiCr (which acts as a binder), 20% Cr₂O₃, 10% Ag, and 10% BaF₂ or CaF₂.

For more information: Michael Trzcinski, Battelle/Great Lakes Industrial Technology Center, GLITeC, 20455 Emerald Parkway

Dr. SW, Suite 200, Cleveland, OH 44135; tel: 216/898-6434; e-mail: trzcinski@battelle.org; Web site: www.glitec.org.

Flame Sprayed Alumina Builds Tougher Composites

A method for preparing aluminate glasses and glass-ceramic composites that opens up new possibilities for generating mechanically strong structural composites and high-hardness coatings has been developed by researchers at the University College London, U.K. The process is designed to toughen brittle materials against fracture.

The flame-spray technique is based on micrometer-size beads of aluminate glass that are formed by feeding oxide powder into a high-temperature hydrogen-oxygen flame and then quenching it. The resulting glassy beads are then sintered into bulk glasses. Further heating produces toughened hard ceramics with the

nanocrystalline alumina-rich phases dispersed throughout the matrix.

According to the researchers, the problem has been that aluminate glasses form only within a narrow range, and no one has yet prepared a bulk glass from pure alumina liquid by standard melt-quenching techniques. However, the flame-spray method yields glassy microspheres containing more than 80% alumina.

The fracture-resistant aluminas are ideal for service in high-temperature, chemically reactive environments such as gas turbines, protective tiles on space probes, and supports for catalytic reactor systems. The method allows the formation of such complex dense shapes by preparing the materials in an amorphous form, such as glass, and then devitrifying them.

Contact: Dr. Paul F. McMillan, University College London, London WC1H0AJ U.K.; tel: 4420-7679-4610; e-mail: p.f.mcmillan@ucl.ac.uk; Web: www.ucl.ac.uk.

News from NASA

Progress in Fabrication of Rocket Combustion Chambers by VPS

Several documents in a collection describe aspects of the development of advanced materials and fabrication processes intended to enable the manufacture of advanced rocket combustion chambers and nozzles at relatively low cost. One concept discussed in most of the documents is the fabrication of combustion-chamber liners by vacuum plasma spraying (VPS) of an alloy of 88Cu/8Cr/4Nb (numbers indicate atomic percentages)—a concept that was reported in “Improved Alloy for Fabrication of Combustion Chambers by VPS” (MFS-26546), *NASA Tech Briefs*, Vol 23 (No.1), Jan 1999, p 50. Another concept is the deposition of graded-composition wall and liner structures by VPS in order to make liners integral parts of wall structures and to make oxidation-and thermal-protection layers integral parts of liners: The VPS process is started at 100% of a first alloy, then the proportion of a second alloy is increased gradually from 0 as deposition continues, ending at 100% of the second alloy. Yet another concept discussed in one of the documents is the VPS of oxidation-protection coats in the forms of nickel-and-chromium-containing refractory alloys on VPS-deposited 88Cu/8Cr/4Nb liners.

This work was done by Richard R. Holmes of Marshall Space Flight Center and Timothy N. McKechnie of Plasma

Processes. This invention is owned by NASA, and a patent application has been filed. For further information, contact Sammy Nabors, MSFC Commercialization Assistance Lead, at sammy.a.nabors@nasa.gov. Refer to MFS-31267.

Composite-Material Tanks with Chemically Resistant Liners

Lightweight composite-material tanks with chemically resistant liners have been developed for storage of chemically reactive and/or unstable fluids—especially hydrogen peroxide. These tanks are similar, in some respects, to the ones described in “Lightweight Composite-Material Tanks for Cryogenic Liquids” (MFS-31379), *NASA Tech Briefs*, Vol 25 (No. 1), Jan 2001, p 58; however, the present tanks are fabricated by a different procedure and they do not incorporate insulation that would be needed to prevent boil-off of cryogenic fluids.

The manufacture of a tank of this type begins with the fabrication of a reusable multisegmented aluminum mandrel in the shape and size of the desired interior volume. One or more segments of the mandrel can be aluminum bosses that will be incorporated into the tank as end fittings.

The mandrel is coated with a mold-release material. The mandrel is then heated to a temperature of about 400 °F (200 °C) and coated with a thermoplastic liner material to the desired thickness [typically, ~15 mils (~0.38 mm)] by ther-

mal spraying. In the thermal-spraying process, the liner material in powder form is sprayed and heated to the melting temperature by a propane torch and the molten particles land on the mandrel.

The sprayed liner and mandrel are allowed to cool, then the outer surface of the liner is chemically and/or mechanically etched to enhance bonding of a composite overwrap. The etched liner is wrapped with multiple layers of an epoxy resin reinforced with graphite fibers; the wrapping can be done either by manual application of epoxy-impregnated graphite cloth or by winding of epoxy-impregnated filaments. The entire assembly is heated in an autoclave to cure the epoxy. After the curing process, the multisegmented mandrel is disassembled and removed from the inside, leaving the finished tank.

If the tank is to be used for storing hydrogen peroxide, then the liner material should be fluorinated ethylene/propylene (FEP), and one or more FEP O-ring(s) should be used in the aluminum end fitting(s). This choice of materials is dictated by experimental observations that pure aluminum and FEP are the only materials suitable for long-term storage of hydrogen peroxide and that other materials tend to catalyze the decomposition of hydrogen peroxide to oxygen and water.

Other thermoplastic liner materials that are suitable for some applications include nylon 6 and polyethylene. The processing temperatures for nylon 6 are lower than

those for FEP. Nylon 6 is compatible with propane, natural gas, and other petroleum-based fuels. Polyethylene is compatible with petroleum-based products and can be used for short-term storage of hydrogen peroxide.

This work was done by Thomas K. DeLay of Marshall Space Flight Center. For further information, access the Technical Support Package (TSP) free on-line at www.techbriefs.com/tsp under the Materials category. 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, Marshall Space Flight Center, tel: 256/544-0021. Refer to MFS-31401.

Manufacturing High-Quality Carbon Nanotubes at Lower Cost

A modified electric arc welding process has been developed for manufacturing high-quality batches of carbon nanotubes at relatively low cost. Unlike in some other processes for making carbon nanotubes, metal catalysts are not used and, consequently, it is not necessary to perform extensive cleaning and purification. Also, unlike some other processes, this process is carried out at atmospheric pressure under a hood instead of in a closed, pressurized chamber; as a result, the

present process can be implemented more easily. Although the present welding-based process includes an electric arc, it differs from a prior electric-arc nanotube-production process. The welding equipment used in this process includes an AC/DC welding power source with an integral helium-gas delivery system and circulating water for cooling an assembly that holds one of the welding electrodes (in this case, the anode).

The cathode is a hollow carbon (optionally, graphite) rod having an outside diameter of 2 in. (~5.1 cm) and an inside diameter of 5/8 in. (~1.6 cm). The cathode is partly immersed in a water bath, such that it protrudes about 2 in. (about 5.1 cm) above the surface of the water. The bottom end of the cathode is held underwater by a clamp, to which is connected the grounding cable of the welding power source.

The anode is a carbon rod 1/8 in. (~0.3 cm) in diameter. The assembly that holds the anode includes a thumb-knob-driven mechanism for controlling the height of the anode. A small hood is placed over the anode to direct a flow of helium downward from the anode to the cathode during the welding process. A bell-shaped exhaust hood collects the helium and other gases from the process. During the process, as the anode is consumed, the height of the anode is adjusted to maintain an anode-to-cathode gap of 1 mm.

The arc welding process is continued until the upper end of the anode has been lowered to a specified height above the surface of the water bath. The process causes carbon nanotubes to form in the lowest 2.5 cm of the anode. It also causes a deposit reminiscent of a sandcastle to form on the cathode. The nanotube-containing material is harvested. The cathode and anode can then be cleaned (or the anode is replaced, if necessary) and the process repeated to produce more nanotubes.

Tests have shown that the process results in ~50% yield of carbon nanotubes (mostly of the single-wall type) of various sizes. Whereas the unit cost of purified single-wall carbon nanotubes produced by other process is about \$1000/g in the year 2000, it has been estimated that for the present process, the corresponding cost would be about \$10/g.

This work was done by Jeanette M. Benavides and Henning Lidecker of Goddard Space Flight Center. For further information, access the Technical Support Package (TSP) free on-line at www.techbriefs.com/tsp under the Manufacturing category.

This invention has been patented by NASA (U.S. Patent No. 6,114,995). Inquiries concerning nonexclusive or exclusive license for its commercial development should be addressed to the Patent Counsel, Goddard Space Flight Center; tel: 301/286-7351. Refer to GSC-14601

People in the News



Dr. Robert C. Tucker, Jr., (left), presenting to Daryl E. Crawmer



Dr. Pierre Fauchais



Dr. Roland D. Seals

Three ASM TSS Members Become ASM Fellows

ASM International inducted 31 new members into the 2004 Class of Fellows during the Annual Awards Dinner that took place on Oct 19, 2004 in Columbus, OH. Of those 31 fellows, three were ASM Thermal Spray Society Members.

Certificates were presented by ASM International immediate Past President Dr. Robert C. Tucker, Jr.

Mr. Daryl E. Crawmer, Director of Technology, Thermal Spray Technologies, Inc., was recognized for "Advancing the Industrial Development and Application of Thermal Spray Technologies and Coatings."

Dr. Pierre Fauchais, Professor, Faculte des Sciences, University of Limoges, was

recognized for "Significant Contributions to Plasma Processing of Materials, and for Thermal Spray Processing Technology."

Dr. Roland D. Seals, Senior Staff Scientist, Oak Ridge Y-12 National Security Complex, was recognized for "Innovative Research and Development in Advanced Manufacturing Processes Based on Surface Engineering and Thermal Spray Technologies."