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News From Institutes and Research Centers Around the World

This column is a forum to inform the thermal spray community on current activities in institutes and research centers active in the field of the thermal spray. Research efforts carried out in these organizations are oftentimes the starting point of significant developments of the technology that will have an impact on the way coatings are produced and used in industry. New materials, more efficient spray processes, better diagnostic tools, and clearer understanding of the chemical and physical processes involved during spraying are examples of such developments making possible the production of highly consistent performance coatings for use in more and more demanding applications encountered in the industry.

This column includes articles giving an overview of current activities or a focus on a significant breakthrough resulting from research efforts carried out in institutes and research centers around the world. If you want to submit an article for this column, please contact: Dr. Jan Ilavsky, UNICAT, APS Bldg 438E, Argonne National Laboratory, 9700 S. Cass Ave., Argonne, IL 60439; tel: 630/252-0866; fax: 630/252-0862; e-mail: ilavsky@aps.anl.gov.

High Temperature and Plasma Laboratory

The High Temperature and Plasma Laboratory (University of Minnesota), founded in the late 1950s, established its reputation under the 36-year directorship of Professor Emil Pfender. The central theme of the research has always been fundamental studies in plasma technology, including plasma-solid and plasma-liquid and plasma-particle interaction, arc electrode effects, plasma instabilities, and influence of fluid dynamics on plasma behavior. Over the past 25 years, an increasing effort has been directed toward the development of plasma processes, including plasma coating and chemical vapor deposition processes, plasma synthesis of nanosize particles, and plasma treatment of hazardous substances. The diversity of the applications on which the authors are working allows considerable synergy to exist between the different projects.

The efforts in plasma spraying have concentrated on analyzing and controlling plasma torch performance and on developing new types of coatings requiring processing under special conditions. For spray torch characterization, the authors have concentrated in recent years on characterizing and controlling the instabilities encountered during the operation of a spray torch. These instabilities result in significant variations of plasma jet properties on a time scale equal to the residence time of a spray particle in the jet, thus affecting coating reproducibility. In particular, the authors have developed arc voltage waveform analysis techniques and sound spectrum analyses as methods for sensing abnormal operating conditions and have correlated these conditions to spray performance. Furthermore, the authors have investigated the use of shrouds to minimize the effects of the jet instabilities. Presently, they are working on a detailed description of the physical causes of these instabilities using diagnostics and three-dimensional modeling. In the development of new coatings, they have concentrated on using the flexibility offered by the Triple Torch Plasma Reactor (TTPR) in which the feed material is injected into the region where the jets from three torches meet—a kind of central injection coating process. One example of coating processes developed is the successive deposition of the three layers making up a solid-oxide fuel cell—a cermet anode consisting of a porous yttria-stabilized zirconia (YSZ) and nickel layer with graded composition, followed by a 99.5% dense YSZ layer serving as the dielectric electrolyte, and finally a porous lanthanum strontium manganite layer serving as the cathode. All three layers were deposited sequentially in the TTPR at high rates. Another ongoing project using this reactor is the development of novel multilayer thermal barrier coatings.

Fig. 1 HTPL plasma spray laboratory

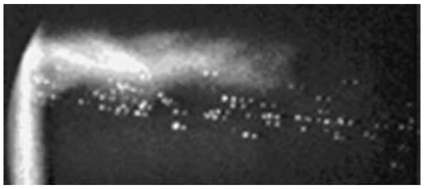

Fig. 2 High-speed image of unstable plasma spray jet with spray particles

The wire arc spray research has concentrated on determining the effects of the fluid dynamics on the metal droplet formation, and as a consequence on the size distribution and the trajectories. Again, a combination of flow visualization and Schlieren photography, high-speed videography with more than 30,000 frames per second, voltage trace analysis, and sound power spectrum analysis have been the authors' primary tools, together with particle trajectory analysis using various instruments, e.g., the widely used DPV 2000. The modeling effort included a complete initial model of the wire arc spray process consisting of several submodels, a three-dimensional arc-in-cross flow model among them. Coating analysis focused on determining porosity, oxide content, bond strength, and interface characterization. In their most recent efforts, the authors have developed nozzle and shroud geometries that allow more

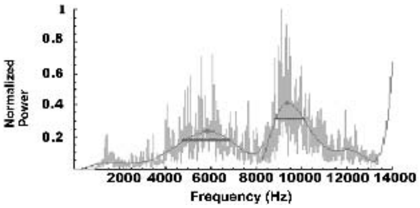

Fig. 3 Spectrum of microphone signal as indication for torch anode condition. Source: Z. Duan et al., *J. Therm. Spray Technol.,* Vol 9 (No. 2), 2000, p 225-234

Fig. 4 High-speed image of the metal droplet formation during wire arc spraying. Source: N. Hussary and J. Heberlein, *J. Therm. Spray Technol.,* Vol 10 (No. 4), 2001, p 2972-2978

controlled spray droplet beam divergence and coating properties. Included in this effort has been the development of a single-wire arc spray torch allowing the deposition of dense coatings in line widths of 3 to 5 mm. Also, a detailed study of the droplet formation and acceleration process is being performed.

Three spray booths are available, in addition to the two controlled-atmosphere/vacuum chambers for the two TTPRs. Several different power supplies allow the study of the influence of this component of a spray system. Among the different plasma torches used in the authors' laboratory, several are of their own design. A vast array of plasma diagnostic equipment is available, including a laser scattering system, several spectroscopy systems and enthalpy probe systems, high-speed video systems, and the necessary image analysis software. In-flight particle analysis can be studied with a DPV 2000, a Stratonics ThermaViz system, or a LaserStrobe Vision system. Coating analysis is being performed in the Surface Characterization Facility for most standard testing and in collaboration with the Chemical Engineering and Materials Science Department for specialized tests.

Among the related projects, a major effort exists in studying the formation and control of nanosized particles over a wide range of pressures and for different materials. In a particular project, the Hypersonic Particle Deposition (HPPD) project, nanosize particles are formed in a supersonic nozzle in front of a plasma torch from chemical precursors injected into the plasma stream. The nanosize particles achieve such high velocities (on the order of 3000 m/s) that inertial deposition on a substrate in front of the nozzle is possible, resulting in a very hard coating of SiC or TiC. Adding an aerodynamic lens between the nozzle and the substrate allows the formation of a collimated nanoparticle beam with a width of 30-50 μm and the deposition of features with corresponding sizes. High-rate vapor deposition processes predominantly of superhard materials, e.g., diamond, boron carbide, and composites, supplement the other coating efforts.

Another project involves fundamental studies of the chemically reacting boundary layer during thermal plasma chemical vapor deposition (TPCVD) of nonoxide ceramics such as SiC and boron carbide, deposited using an RF thermal plasma. In this study a molecular beam mass spectrometry (MBMS) system is used to measure the mole fractions of chemical species in gas that is sampled through a small orifice in the film growth substrate. This coupled TPCVD/MBMS system has proven capable of providing quantitative measurements of radical species at concentrations as low as 0.1 ppm. These measurements, together with kinetic modeling and film growth experiments, are providing a detailed picture of the physical and chemical processes that govern film growth during TPCVD.

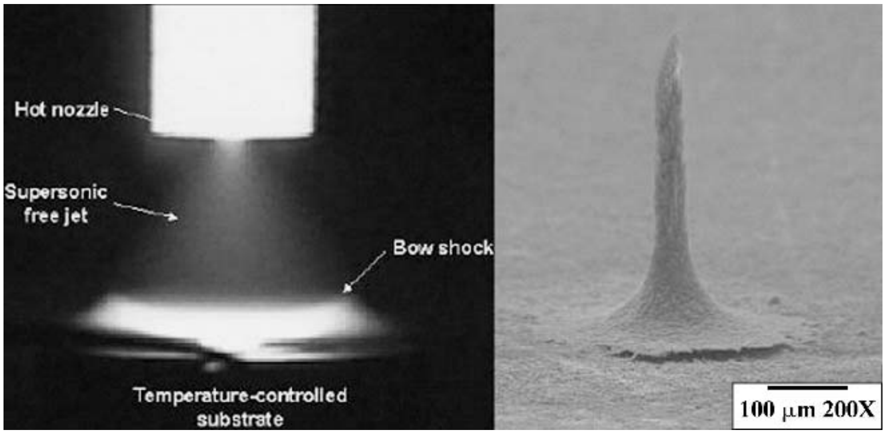

Fig. 5 Hypersonic plasma particle deposition process (left) and a 30 μm diameter needle deposited with a collimated nanoparticle beam

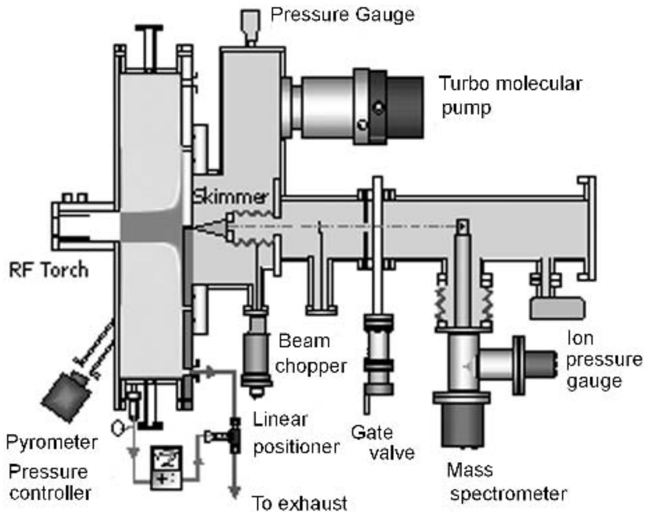

Fig. 6 TPCVD reactor and MBMS system

A further project on nanoparticle synthesis is being conducted at the HTPL under the auspices of the Center for NanoEnergetics Research at the University of Minnesota. Because of their high specific surface area, nanoparticles have intrinsically high chemical reactivity and are thus of interest for applications such as solid-fuel propellants. In this project, aluminum nanoparticles are synthesized in a thermal plasma expansion. A major focus of the project is to develop methods for coating and passivating nanoparticle surfaces so that their reactivity can be retained until needed. Two methods are being explored to accomplish this. In the first, the nanoparticle-laden plasma jet is opposed by a cold counterflow, which suppresses further particle growth and coagulation, and which can also deposit a thin coating on the particles by the addition of appropriate reactants in the counterflow. In the second approach, the particles will pass through a chamber where they are coated by a polymer by exposing them to radiation from ultraviolet (UV) lamps in the presence of a hydrocarbon such as acetylene.

As already mentioned, a significant modeling effort is pursued in parallel to the authors' experimental characterization to allow the development of a detailed understanding of the process physics and chemistry. Specific models include plasma-particle heat and momentum exchange, particle nucleation and growth, and several different codes for describing the plasma fluid dynamics. This modeling effort is pursued in collaboration with the Minnesota Supercomputer Institute.

Funding for the research is derived from several grants from NSF, DOE, NASA, and from collaborative projects with industry. There are presently four professors within the laboratory, including Prof. J. Heberlein, S. Girshick, U. Kortshagen, and Prof. Emeritus E. Pfender, two postdoctoral associates, 20-24 graduate students, and several visiting scholars. Numerous collaborations exist with other professors within the University and with groups at other universities. The laboratory occupies about 8474 square feet in the new and old Mechanical Engineering buildings.

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Spray Drying and Gas Atomization at LERMPS to Develop New Sprayable Materials for Advanced Material Performances

LERMPS (Laboratoire d'Etudes et de Recherches sur les Matériaux, les Procédés et les Surfaces), the surface engineering laboratory of the Université de Technologie de Belfort-Montbéliard (UTBM, Technological University of Belfort-Montbéliard), France, is an academic research team whose complementary activities concern:

- thermal spraying (plasma, high-velocity oxyfuel, arc, flame),
- physical vapor deposition (magnetron sputtering, reactive sputtering, etc.), and
- precursor materials and feedstock (powders, targets, etc.).

With its 70 members, among them 22 Ph.D. students, LERMPS developed its facilities through the years for the development of feedstock powders. Today, this facility comprises principally a spray dryer for ceramic and composite powders, a gas atomizer dedicated to high-purity metallic alloy powder, and several dedicated apparatuses (i.e., sintering furnaces, sieving machines, particle size analyzer, etc.).

Development of New Generations of Powders by Spray Drying

Powder Architecture

Among the numerous parameters that influence coating quality, the powder morphology is of prime importance. It is noteworthy that spray drying is probably the most versatile powder-processing technique. This method allows agglomerating any kind of small particles in spherical shapes by means of an organic binder. Powders prepared in this way can be submitted to further densification by sintering in a furnace or by plasma or flame treatment.

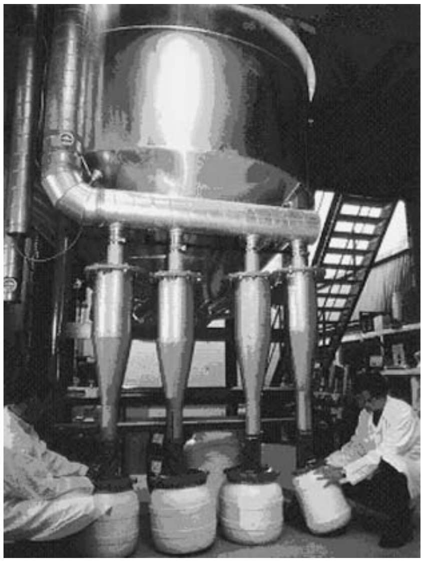

Fig. 1 Spray dryer apparatus designed and put into operation at LERMPS

Therefore, in order to prepare specific thermal spray powders, particularly composite powders, a spray dryer apparatus has been designed in the laboratory (Fig. 1). The spray drying process consists of a spraying slurry containing finely dispersed particles of the materials to be agglomerated, organic compounds, and water. A pump feeds the slurry into an atomizing nozzle designed with a special geometry. The atomized slurry is thrown into a large cyclonelike chamber in a countercurrent stream of heated air. The moisture contained in the droplets evaporates during their flight in the chamber. The resulting solid particles are collected at the bottom of the chamber and are separated from the gas in cyclone collectors. Spray dried granules have a large variety of shapes; from uniform solid spheres that are regarded as ideal granules in most cases to elongated, pancake, donut-shaped, needlelike, or hollow granules. By varying processing parameters, granules of these different shapes can be produced. Powder morphology, chemical composition, size, density, flowability, and friability are the most important physical and chemical characteristics regarding their use as feedstock material in the thermal spray processes.

Spray Drying

To develop new compositions or new morphologies of powders, two steps have to be managed simultaneously: on the one

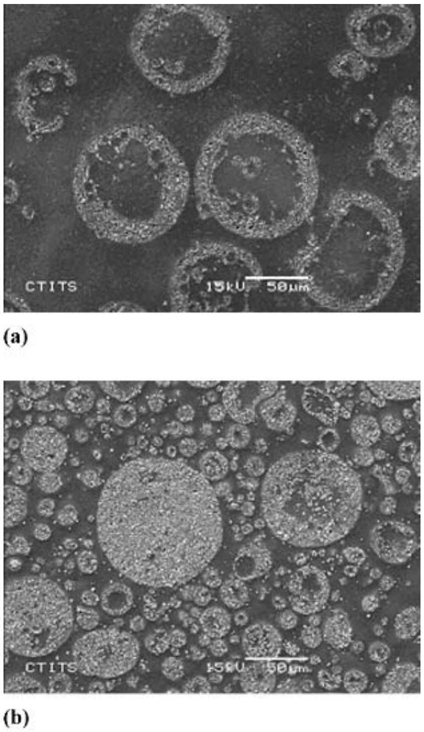

Fig. 2 Polished cross sections of spray dried alumina granules from (a) a dispersed suspension (pH 9, 0.1 wt.% PAA, 15 wt.% latex) and (b) a flocculated slurry (pH 4, 0.1 wt.% PAA, 15 wt.% latex)

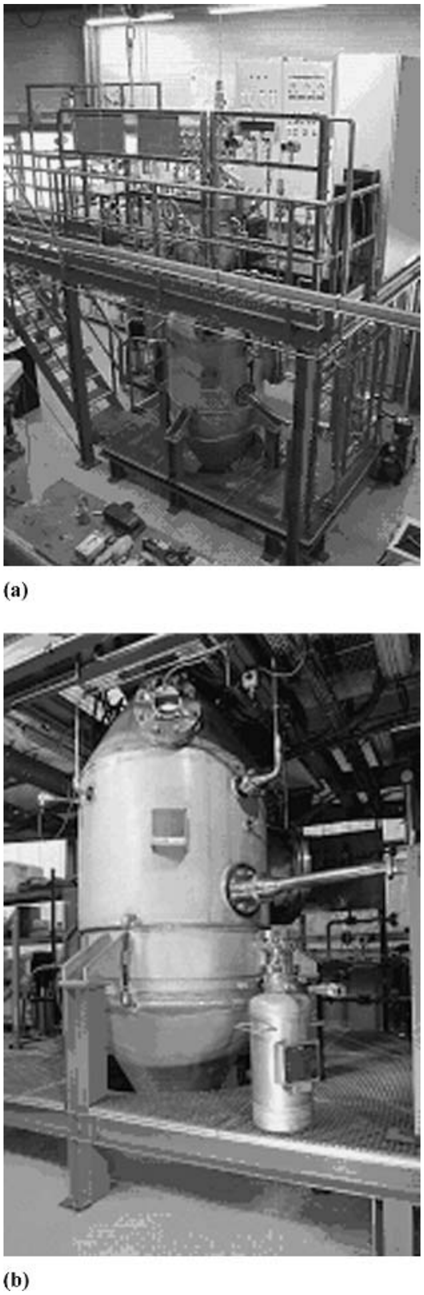

Fig. 3 Atomizing facility in the LERMPS

hand, the composition of the slurry and its drying stage and on the other hand the technique and conditions of the slurry atomization. Indeed, the process variables determine to a degree the powder particles morphology and size. In particular, the reactor is equipped with a two-fluid nozzle using a pressurized air jet to break up the slurry into droplets. As a consequence, the design of the nozzle, the degree of feed aeration, and the concentration of the slurry are major process factors. For example, a performance characterization study has been conducted on Al_2O_3 powders and has shown that the average powder grain size is increasing with the nozzle diameter and decreasing with the air-flow rate. Concerning the grain size distribution, this is the interaction between the air-flow rate and the nozzle diameter that has the most important influence. More recent works focused on the atomized droplets flow characteristics. It was shown particularly that the design and diameter of the atomization nozzle influence the width and concentration of the droplet flow. Straight-designed nozzles lead to narrow and concentrated jet in contrast with conical designs. Besides using optical diagnostics, it was demonstrated that the speed distribution is flatter and the speed value is lower with a conical design than the ones achieved with a straight nozzle design.

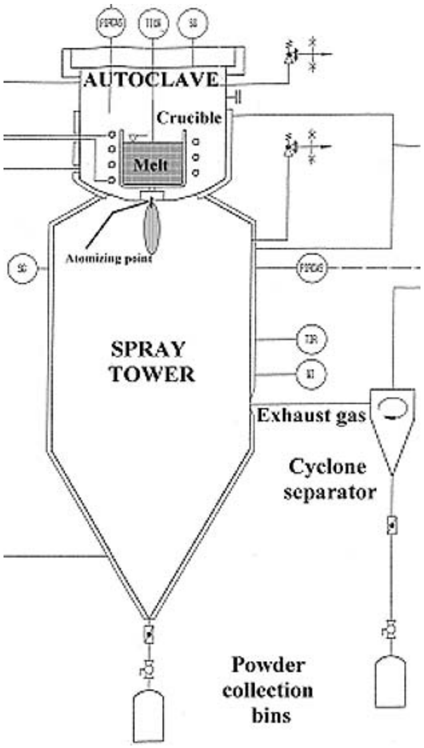

Fig. 4 Synoptic of the LERMPS atomizing facility

Controlled Hollow Sphere Spray Powders

Previous results concern mainly the parameters that have an influence on the powder size, but the key point in using the spray drying process is to be able to change the morphology and composition of the produced powders. These characteristics closely depend on the physical and chemical properties of the slurry combined with the drying stage, as has now been demonstrated. The idea behind this was that the state of dispersion of the slurry is the major characteristic controlling the dried granule shape. In a first step, studies were conducted in the laboratory to determine the influence of the formulation (i.e., solids loading, pH, ionic strength, amount of dispersant or type of binder, etc.) on the stability and homogeneity of slurries using sedimentation tests (i.e., sedimentation kinetics and long term sedimentation value), electrophoresis mobility measurements (i.e., zeta potential data), and supernatant total organic carbon analyses (i.e., dispersant adsorption isotherms). For instance, yttria partially stabilized zirconia (Y-PSZ) and alumina slurries showed similar behaviors versus interactions between particles and organic additives (dispersant and binder) with pH, and sedimentation experiments appeared to be appropriate and sufficient to assess the dispersion state. Thus, drying simulated experiments based on the drying of a single suspended droplet have been performed with various slurries formulations achieved by changing the pH, the amount of dispersant, and the type of binder. Two major granule shapes have been identified after the drying tests: a solid morphology and a hollow one with adjustable shell thickness. A quantitative correlation was established between the state of dispersion (i.e., the long term sedimentation value, RS) and the granule shape. This clearly shows that low RS levels (below 0.6 for zirconia slurries or 0.7 for alumina slurries), which correspond to dispersed slurries as confirmed by transmission optical images, provide dried droplets which are hollow spheres (Fig. 2). In contrast, high RS values, which are obtained with flocculated slurries, lead to solid dried granules. Besides, a linear relation was established between the shell thickness of hollow granules and the value of the sedimentation ratio. Indeed, the lower the RS the best dispersed the slurry and the thinner the shell. Finally, drying mechanisms have also been discussed and established. At the beginning, the surface temperature of the droplet quickly increases and the surface moisture begins to evaporate, accompanied by shrinkage. If an outer crust forms, which is the case with dispersed slurries, the shrinkage stops and at the same time the internal pressure due to moisture increases. Due to the porosity of the shell, the pressure can be released and a hollow sphere is formed. For flocculated slurries, the drying process is controlled by the continuous outward diffusion of moisture. As no crust is formed, the shrinkage proceeds continuously and can be important. Spray dried powders were produced on the basis of these results and showed that the single-droplet drying test is an efficient test to predict the morphology of the spray-dried powder.

Although the spray drying method has been known and used since more than 100 years, especially in the food and chemical industries, now the end-product characteristics can be predicted as far as monoceramic powders are concerned; however, a special effort still has to be dedicated to the thermal spray community demand for new composite powders (ceramic-ceramic or polymer-ceramic).

High-Purity Spray Metallic Alloy Powders by Gas Atomization: Nanoval Process

Gas Atomization

A metallic powder atomizing in neutral atmosphere (argon) facility was erected in May 2003 at the original LERMPS facility where thermal spraying is performed. This tower is shown in Fig. 3,and a simplified synoptic is displayed in Fig. 4.

The facility consists in its main part of an autoclave head, a spray tower, a gas distribution system and an exhaust gas system (cyclone separator). In the autoclave head are the insulated crucible heated by an induction coil, and the special atomizing unit, containing mainly the Laval nozzle and the melt nozzle. This is where the Nanoval Process (European Patent No. EP 0 220 418 B1, Gerking Lüder, Oct. 25, 1989, and United State Patent No. US 4,822,267, Walz Alfred, April 18, 1989) occurs. It was appeared at the beginning of the 1990s and has five characteristics: atomizing gas, batch weight, particle size distribution, efficiency and atomized materials. Table 1 summarizes them.

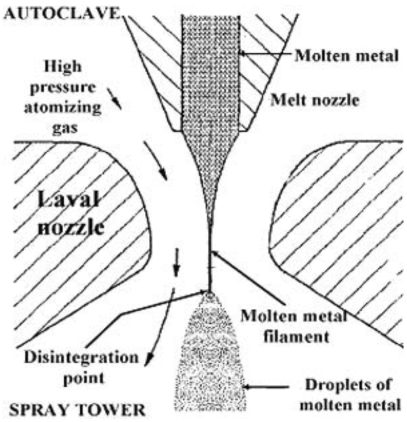

Fig. 5 De Laval nozzle with melt in the center and atomizing gas around it

The atomizing facility was developed thanks to national funds via Inter-Ministerial Commission for Land Planning (Comité Inter-Ministeriel pour l'Aménagement du Territoire, or CIADT) and to European Regional Development Funds (Fonds Européens de Développement Régional, or FEDER). LERMPS Laboratory is a member of the institute of the surface treatment of Franche-Comté (ITSFC) in France.

Atomization Mechanisms

The atomization is realized in a round Laval nozzle flow, which is positioned underneath the melt out-flow nozzle (Fig. 5).

The gas flows around the melt nozzle and the melt monofilament into the Laval nozzle. The gas flow, which is laminar, is steadily accelerated and reaches sonic speed in the throat of the Laval nozzle, its narrowest cross section.

The melt monofilament is deformed to an ever-decreasing diameter. When surface tension pressure in the liquid stream surpasses the outer pressure, bursting of the melt can happen. A necessary condition for this effect is a very steep increase of gas flow acceleration. In supersonic gas dynamics, this is accompanied by a strong pressure decrease leading to the stream disintegration. After the melt monofilament disintegration, the formed particles are still in the state liquid. Surface tension can form spheres. This constitutes the second characteristic feature of the Nanoval process. Located in the spray tower, the liquid metal droplets solidify by convective exchange with the cold gas and thus made powder (Fig. 6).

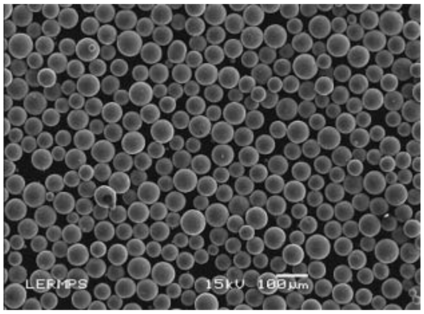

Fig. 6 As-atomized powder

High Process Efficiency

Most of the elaborated powder is collected in a powder collection bin underneath the spray tower. The gas and entrained powder flows out of the spray chamber into a cyclone separator. Here, most of the fine powder is removed out of the gas and collected in another powder collection bin. This allows keeping nearly all the atomized powder in order to improve the facility efficiency.

Controlled Particle Size Distribution

The third characteristic of this process is a better control of the particle size distributions with a range moving to lower sizes. Figure 7 displays examples of particle size distributions obtained with the facility.

Mean particle diameters (D_{50}) between 5 and 45 μm were achieved depending on the used metal and autoclave pressure. Metals with higher specific density achieved lower D_{50} values than those measured for less dense metals.

Narrow Particle Size Distribution

This process achieves values of $1.3 < (D_{84}/D_{50}) < 1.8$, whereas all others lie between 2.3 and 3.5. In the latter case, the particle size distribution is so wide that it requires a mechanical sieving into several fractions, which makes it difficult to promote some of them.

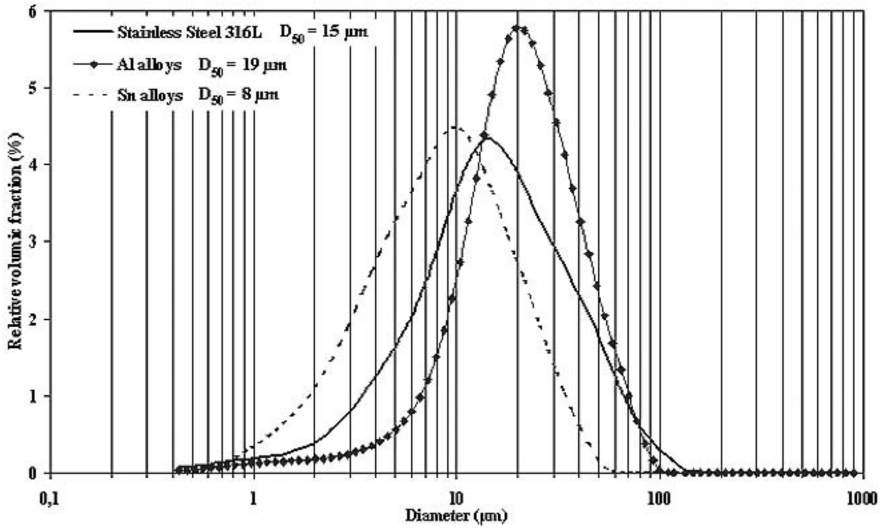

Fig. 7 Example of powder particle size distribution obtained in the LERMPS atomizing plant

Table 1 Main Characteristic of the LERMPS Gas-Atomizing Facility

		Average Particle Size Distribution, µm				
Atomizing Gas	Batch Weight, kg	D_{50}	D_{84}/D_{50}	General Range	Efficiency	Atomized Materials
Argon	1-70	5-45	1.3-1.8	1-100	>95%	Fe, Ni, Cu, Co, and Al alloys

Flexible System

As the spectrum of the particle distribution moves to lower sizes, the size of the formed droplets in the chamber is smaller. Then they solidify more quickly, thus requiring a smaller-size atomization facility. In fact, the facility located in LERMPS is less than 4 m high. In addition, some small powder batches can be elaborated, it is the fifth characteristic. The range of the batch can vary between 1 and 70 kg according to the raw material density, though in classical processes of gas atomization using a large facility, only large amounts of powder (several tons/batch) can be elaborated.

Feedstock Suitable for Several Technologies

Iron-, nickel-, copper-, cobalt-, or aluminum-base metallic powders processed with the LERMPS Atomizing Facility can be used for the following technologies:

- metal injection molding processes (MIM-PIM),
- thermal spraying,
- memory alloys (i.e., powder of different compositions for parts showing the mechanical or thermal memory effect),
- laser sintering, and
- rapid prototyping (stainless steel 316L powder or other steel or other metallic element for a new prototyping process able to produce full dense parts).

Fig. 8 Molten metal jet at the exit of the atomizing nozzle

Research on Process Operating Parameters

Moreover, this facility is equipped with special view ports to diagnose the molten metal jet in the spray tower (Fig. 8). Purpose is to make investigations regarding how the monofilament explodes at the atomizing point. One can also observe both temperature and particle speed like diagnostic experiences made with thermal spraying processes. At the moment, a Ph.D. student is in charge of this subject to develop the diagnostics in the LERMPS Atomizing Facility.

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Industrial News

Corona-Treating Rolls

In the past, there were two types of corona-treating systems: covered-roll and bare-roll systems. Since then time, ceramic-coated ground rolls found more use because they provide more power than their predecessors and also eliminate common problems associated with covered rolls. While the cost of a new silicone roll covering is not great, many companies realize there are more costs associated with replacing them than just the cost of the new sleeve. The silicone-covered roll could become damaged from either overexposure to corona or because it was cut with a knife. If the damage is severe enough it will cause a high-voltage trip, which will result in shutting down the line, most likely during an important run, and replacing the roll covering. If the damage is not enough to cause a high-voltage trip, the substrate could be experiencing pinholing or backside treatment. These two problems are often difficult to detect and will certainly compromise the quality of the product, and this problem may not be found until the customer complains. By this time the production line may have produced several hundred thousand feet of substandard material resulting in excessive scrap, customer complaints, and lost production time during shutdown to replace the roll covering. So, the costs can quickly add up to more that just the cost of the roll covering.

Universal-roll treaters can mitigate these problems. Ceramic-covered rolls, conductive ceramic-coated rolls, and universal ceramic-coated rolls are described:

- **Ceramic-covered rolls:** These rolls are covered with a thick ceramic covering (0.06-0.10 in.) that is applied via a plasma spray technique. This roll covering replaces the rubber or epoxy coverings used with metal electrodes to provide a dielectric buffer between the metal electrode and the roll covering, allowing the air to ionize to create the corona in the air gap. Ceramic-roll covering is widely used because of its high dielectric strength and resistance to physical damage. This type of electrode/roll combination cannot be used to treat conductive films.
- **Conductive ceramic-coated rolls:** Conductive ceramic is a thick coating (0.002-0.005 in.) applied via a plasma spray technique. This coating is employed on bare-roll systems where ceramic electrodes are used and protects the ground roll from oxidation (corrosion). This covering replaces chrome or electrodeless nickel plating, which are porous and allow oxidation between the roll core and the plating. Conductive ceramic-roll stations can be ideal for metallized film and foil. A conductive ceramic-coated ground roll in a bare-roll system is recommended when treating metal foils or the nonconductive side of conductive films. The conductive ceramic coating ensures a conductive path to ground from the conductive web surface. The primary benefit of the conductive ceramic is to prevent oxidation and other damage to the roll surface.
- **Universal ceramic-coated rolls:** Universal ceramic is also applied to the roll core via a plasma spray process. The universal ceramic coating is nonconductive and also serves as a dielectric covering on the ground roll. This roll coating is used in systems employing ceramic electrodes. Although the universal ceramic coating is nonconductive, it is unlike the ceramic coating used with metal electrodes. In fact, the universal ceramic is not to be used with metal electrodes.

Treating Metallized Films

A universal ceramic-coated roll system can be used to treat metallized films or foils. Because both the high-voltage electrode and the ground electrode (ground roll) are covered with a dielectric, the corona will be established in the air gap between the electrode and metallized film or foil. However, it is important that the metallized surface of the film be in contact with a clean and grounded idler roll. This idler roll can be covered with conductive ceramic to prevent oxidation and provide a ground path from the conductive film surface.

The advantages of using a universal ceramic-coated roll system versus a bare-roll system are:

- superior treatment levels,
- elimination of film wrinkling or puckering,
- elimination of backside treatment, and
- elimination of pinholing on the metallized surface.

Conclusion

The universal roll can be superior when compared to silicone-covered roll. While the initial cost of a ceramic-covered roll is greater than a silicone-covered roll, it can cost less over the life of the station. It is important to note that in some cases the initial costs can be very close. The higher-efficiency ceramic-covered roll needs less power to meet the same treat requirements, thereby reducing the cost of the power supply required.

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Fuel Cells to Advance Zero-Emissions Energy; New Research Will Help Fuel Cells Move to Commercial Viability

Secretary of Energy Spencer Abraham announced a new phase of fuel-cell research designed to hasten the wider availability of zero-emissions energy. Eleven new projects with total value of nearly $4.2 million, including private-sector cost sharing of more than 20%, focus on solving the remaining issues in developing solid-oxide fuel cell (SOFC) systems for commercial use.

Fuel cells are one of the cleanest and most efficient technologies for generating electricity. Since there is no combustion, there are none of the pollutants commonly produced by boilers and furnaces. For systems designed to consume hydrogen directly, the only products are electricity, water, and heat. Fuel cells operate much like a battery, turning oxygen and hydrogen into electricity in the presence of an electrically conductive material called an electrolyte. Unlike a battery, however, fuel cells never lose their charge. As long as there is a constant source of fuel—usually natural gas for the hydrogen and air for the oxygen—fuel cells will generate electricity. Uses and potential uses of fuel cells include on-site electric power for households and commercial buildings; supplemental or auxiliary power to support car, truck, and aircraft systems; power for personal, mass, and commercial transportation; and the modular addition by utilities of new power generation closely tailored to meet growth in power consumption.

Selected by the Department of Energy's (DOE) Solid State Energy Conversion Alliance Program (SECA), the grant-winning projects are focused on developing improvements in fuel-cell materials and performance, as well as attaining target capital costs of less than $400 per kilowatt, all of which will make fuel cells extremely competitive with conventional power generation. Successful scale-up of SECA fuel cells will allow these ultra-high-efficiency power modules to provide pollution-free electricity from zero-emission plants such as FutureGen. The SECA Program is managed by DOE's National Energy Technology Laboratory. Among the projects is grant to University of Connecticut to develop a multilayered composite structure consisting of thin layers of oxidation-resistant metals, porous ceramics, and glasses. The seal structure will be fabricated onto the surfaces of ceramic and metallic SOFC components using low-cost manufacturing methods such as atmospheric plasma spray.

Contact: Jeanne Lopatto, tel: 202/586-4940 or Drew Malcomb, 202/586-5806; U.S. Department of Energy.

McGill Researchers Develop New Carbon Nanotube Production Method

McGill University researchers have developed a new method for producing carbon nanotubes that has great commercial promise. The work of Professor Jean-Luc Meunier and doctoral student David Harbec, both of the Department of Chemical Engineering, is the subject of a patent application, and the findings of their team have been published in the *Journal of Physics D: Applied Physics.*

Carbon nanotubes (CNTs), discovered in 1991, are seamless cylinders composed of carbon atoms in a regular hexagonal arrangement, closed on both ends by hemispherical endcaps. They exhibit remarkable mechanical and electronic properties. Applications include high-strength composites, advanced sensors, electronic and optical devices, catalysts, batteries, and fuel cells.

The current low-volume production methods and high production costs are the limiting factors in the CNT high-growth market. The McGill researchers developed a new method and apparatus to produce CNTs with the possibility of scale-up to large industrial levels that is based on thermal plasma technology. Plasmas form the fourth state of matter after gas, while the term "thermal plasmas" refers to their typical state of almost thermal equilibrium between electrons, ions, atoms, and molecules. Thermal plasmas typically have temperatures between 4000 and 25,000 °C and are created by electric arcs or magnetic induction discharges.

"The use of carbon nanotubes in advanced materials is not only limited by their price, but more importantly by their unavailability in large quantities," notes Prof. Meunier. "This method using thermal plasmas brings production towards industrial levels at megawatt powers, and Quebec is an important player worldwide in thermal plasmas."

Meunier and Harbec are the authors, along with McGill researchers Liping Guo, Raynald Gauvin, and Nadine El Mallah, of the article "Carbon Nanotubes from the Dissociation of C_2Cl_4 Using a dc Thermal Plasma Torch," appearing in the July 14 issue of *Journal of Physics D: Applied Physics.*

McGill University is currently seeking licensees to its patent-pending technology for producing CNTs, and the McGill researchers have just received an Idea to Innovation grant from the Natural Sciences and Engineering Research Council of Canada to help bring their technology closer to market.

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Terolab Services Group in Lausanne, Switzerland, Expands in Germany

Terolab Services (TLS) has taken over with immediate effect the assets of one its competitors, the Gotek Hartstofftechnik in Frankfurt. Terolab Services, created in 1998 by Christopher Wasserman, produces composite elements for wear protection in Switzerland, Germany, France, and Austria. The Group has 200 employees and anticipates to reach sales of Swiss francs 30 million, a sound increase compared to 2003.

Contact: Christopher Wasserman, Terolab Services Management SA, World Trade Center, CP 476, CH-1000 Lausanne 30, Switzerland; tel. +41 21 641 50 22; e-mail: Ch.wasserman@terolabservices.com. Web: www.terolabservices.com.

News from NASA

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, or 0.38 mm) by thermal 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 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. 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, 256/544-0021. Refer to MFS-31401. Excerpted from *NASA Tech Briefs,* Sept 2004.

Snippet of Information

Microwave Atmospheric Plasma

Dana Corp., Toledo, Ohio, has deployed a revolutionary, proprietary technology that harnesses microwave energy to accomplish dozens of processes, including heat treating and coating of metal and ceramic parts.

Microwaves historically have not been used to process metals because metals do not absorb microwaves, and the reflection can damage the microwave source. Dana's microwave plasma technology solves this problem by surrounding the metal with microwave-absorbing plasma at atmospheric pressure.

Although the initial focus is on metal processing and coating, Dana is exploring a variety of nontraditional applications such as exhaust treatment, surface engineering (including decrystallization), formation of carbon nanostructures, and hydrogen production.

News from Professional Societies

Joint e-Memberships Make ASM More International

ASM International has begun "Joint e-Memberships," providing cost-effective electronic benefits of ASM membership to members of other societies.

Following a pilot program with the Institute of Materials Engineering Australasia (IMEA), the first European e-Membership program with the Société Française de Métallurgie et de Matériaux (SF2M) was launched with more than 60 SF2M members participating as of June. ASM is also working to partner with societies in India, China, Mexico, Japan, and the United Kingdom to create additional Joint e-Membership programs.

The cost of Joint e-Membership is a nominal annual fee in addition to their regular national society dues. Benefits include unlimited access to the *ASM Metals Handbook Desk Edition Online* and *Engineered Materials Handbook Online,* archived articles from *Advanced Materials & Processes* and *Heat Treating Progress* magazines, and communication tools for networking with other ASM members. Joint e-Members are also entitled to member discounts on ASM books, online reference collections, and journals, and are encouraged to participate in ASM technical programs, committees, and the ASM awards program.

Contact: Thomas S. Passek, ASM Associate Managing Director; tel: 440/338-5151, ext. 5505; e-mail: Thom.Passek@asminternational.org.

Abrasive Wear Task Group Begins Work on Chemical-Mechanical Polishing Standard

ASTM Committee G02 on Wear and Erosion has committed itself to standardizing the chemical-mechanical polishing process by creating a task group to develop a proposed new standard, tentatively titled "Test Method for Wear and Friction Testing of Polishing Pads, Slurries, and Pad Conditioners in Chemical-Mechanical Polishing (CMP)." This activity, which has been assigned ASTM work item number WK5036, is under the jurisdiction of Subcommittee G02.30 on Abrasive Wear.

Chemical-mechanical polishing has been one of the fastest-growing segments of the semiconductor industry for the past 10 years. However, interest in chemical-mechanical polishing has not been limited to semiconductors—thousands of chemical-mechanical polishers are currently being used in the precision glass, optics, and magnetic media, as well as other high-tech industries.

Despite the popularity of chemical-mechanical polishers, growth of the CMP industry has been limited due to a lack of standardization. For example, there has been no incoming inspection of CMP consumables—such as polishing pads, slurries, and pad conditioners—in the industry, resulting in dramatic process fluctuations. In addition, there has been a lack of test procedures and test equipment to characterize CMP materials.

"The proposed new standard will help the high-tech industry to collaborate in developing the standardized effective test procedures for CMP materials," said Norm Gitis, Center for Tribology, who also noted that all interested parties are invited to join the task group. "The work on the standard has been started, and we are looking for collaborators from various industries and academia."

Contact: Norm Gitis, Center for Tribology, Campbell, CA; tel: 408/376-4040; e-mail: NGitis@cetr.com.

ASTM International Committee E01 Preparing to Implement Performance-Based Methods

At its May 2004 meeting, the executive subcommittee of ASTM Committee E01, Chemical Analysis of Metals, Ores, and Related Materials, unanimously committed to develop and implement performance-based test methods for the quantitative analysis of materials under its jurisdiction. Performance-based test methods define general approaches for sampling, sample preparation, and making measurements on a specified type of material. They set maximum allowable uncertainties for each component of uncertainty of each measured constituent over its validated concentration range. The key criteria of compliance with a performance-based standard test method is the quality of data generated (uncertainty) rather than adherence to procedure.

The new E01 performance-based approach is based on ASTM standards E 691, Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method; E 1601, Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method; E 2165, Practice for Establishing an Uncertainty Budget for the Chemical Analysis of Metals, Ores, and Related Materials, and a proposed new standard, Practice for Designing and Validating Performance Based Test Methods for the Analysis of Metals, Ores, and Related Materials, which is now on ballot. A second proposed new Practice for Implementing Standard Performance

Based Test Methods for the Analysis of Metals, Ores, and Related Materials will be balloted shortly and describes how laboratories will be expected to comply with performance-based standards in a way that is compliant with ISO 17025, General Requirements for the Competence of Testing and Calibration Laboratories.

E01 committee member Dean Flinchbaugh, who presented a workshop on performance-based test methods during the May meeting, says that Committee E01, ASTM International as a whole, the laboratories that use its test methods, and the bodies that accredit laboratories will benefit from the use of performance-based methods. Some of these benefits include: (1) having standard test methods that are more resistant to obsolescence, (2) streamlining test method validation and interlaboratory testing, (3) having more consistent data quality reported from multiple laboratories that use the same test method, (4) expanding procedure flexibility in user laboratories, (5) having more consistent measurement uncertainty information to facilitate product conformity decisions, and (6) simplifying internal and external audits by having fewer procedural details to examine.

Further, Flinchbaugh says the new approach is compatible with existing documents that are used internationally. Examples include compliance with ASTM Practices E 1601 and E 2165, as well as compatibility with existing test methods and proficiency test programs and ISO 17025. The performance-based concepts are also applicable to other ASTM technical committees that write analytical chemistry-based test methods.

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People in the News

Altair Nanotechnologies Appoints New Chief Executive Officer

Altair Nanotechnologies, Inc. (NASDAQ: ALTI), announced that it has appointed Alan J. Gotcher, Ph.D., as its Chief Executive Officer. Gotcher fills the position created by the May 1, 2004 retirement of Dr. William P. Long. Gotcher has been working with Altair as a management consultant since May 2004.

Before joining Altair, Gotcher was Chairman and CEO of Nevada-based InDelible Technologies, Inc., a development stage company that provides secure logistics through covert bar-code marking systems and invisible bar-code reading technologies. Prior to founding InDelible, Gotcher spent 14 years with Avery Dennison in Pasadena, CA.

Previously, Gotcher was Laboratory Director, U.S. Corporate Research and Development, with Raychem Corporation (based in Menlo Park, CA), where he lead the business development teams that created, developed, and commercialized the conductive polymer-based Poly-Switch over-current protection device business.

Gotcher resides in Incline Village, Nevada, and has a Ph.D., Chemistry, and a B.S., Chemistry, from the University of California, Irvine.