In The News

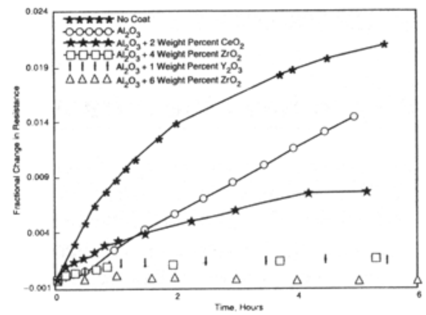
Protective Coats for High-Temperature Strain Gages

Rare-earth oxide additives in alumina reduce oxidation

Lewis Research Center, Cleveland, Ohio

The addition of some rare-earth oxides to the prior alumina (only) coating material increases the maximum service temperature of palladium/chromium-wire strain gages. Previously limited to use at temperatures below 400 °C (750 °F), the Pd/Cr wires can now be used at temperatures up to 800 °C (1470 °F) without excessive drift in electrical resistance.

The oxides used are zirconia (ZrO_2), yttria (Y_2O_3), ceria (CeO_2), and hafnia (HfO_2). The effect of the addition of one of these oxides to the alumina coat is to decrease the oxidation of the wire at high temperature. The protection against oxidation increases with the concentration of the rare-earth oxide, up to a limiting concentration of 6% ZrO_2, 1% Y_2O_3, 2% CeO_2 or 1% HfO_2. Above these concentrations, the rare-earth oxides can react with the Pd/Cr wire. As shown in the figure, the addition of ZrO_2 at 4 to 6 wt% or Y_2O_3 at 1 wt% results in the smallest drift in electrical resistance.



Small amounts of rare-earth oxides in a protective coat reduce the change in electrical resistance of a Pd/Cr wire 25 µm (0.001 in.) in diameter at a temperature of 800 °C (1470 °F). Y_2O_3 at 1 wt% or ZrO_2 at 4 to 6 wt% yield the lowest change in resistance and were selected for use in protective coats on Pd/Cr wire strain gages.

To begin the process of mounting a strain gage, a coat of Ni/Cr/Al alloy 25 to 50 µm (0.001 to 0.002 in.) thick is flame sprayed onto the surface of the strain specimen. Next, an alumina coat 25 to 50 µ (0.001 to 0.002 in.) thick is applied by flame spraying. The wire strain gage is then laid on the alumina-coated surface, and a protective coat of alumina containing zirconia or yttria is flame sprayed over the wire grid and surrounding area to a thickness of 100 to 200 µm (0.004 to 0.008 in.).

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Grinding Out Solutions With Intelligent Abrasive Machining

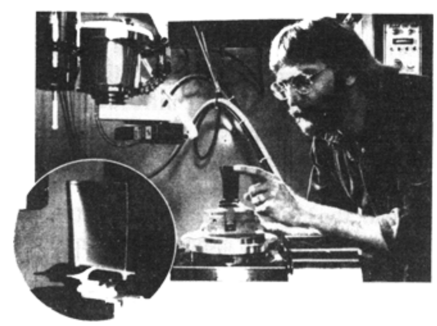
Automated, adaptive process removes grit overspray from jet engine turbine blades

Just as automobile engine performance is improved by minimizing the gap between the piston ring and the cylinder wall, jet engine performance depends on the size of the gap between the tip of a turbine blade and the inside of the engine case, or shroud. If the gap is too large, combustion gases leak past the blades, sapping power. Too small a gap could send a thermally expanding blade right into the shroud during a power increase.

Sandia and Pratt & Whitney, a leading producer of gas-turbine engines for commercial and military aircraft, have entered into a Cooperative Research and Development Agreement (CRADA) to develop an improved process for high-quality turbine blades with minimum air seal leakage. To minimize leakage, Pratt & Whitney now uses an active "clearance control system" in which abrasive-tipped blades actually cut shallow grooves into the engines.

To coat the blade tips with abrasive, Pratt & Whitney applies a silicon carbide grit to the tips and bonds it in place by plasma spraying a nickel alloy around the grit. However, plasma spraying creates overspray—excess alloy extending 1 to 3 mm (0.04 to 0.12 in.) down the side of the blades. This material must be removed carefully to restore the airfoil shape for high performance and to avoid disjoint surfaces (stress risers) that could lead to blade failure in the high-temperature, high-stress environment of an operating jet engine.

"Common practice is to manually remove overspray with a sanding belt," explains Pratt & Whitney development engineer Paul Phaneuf. "But this approach depends on factors such as the operator's skill, interest level, and fatigue." The Pratt & Whitney/Sandia CRADA will focus on developing an automated overspray grinding technique based on Sandia's experience in intelligent manufacturing systems.



Cliff Loucks examines the surface finish after using adaptive machining to grind excess abrasive material from the tip of a turbine blade.

Grinding the overspray off complex contours of blades to tolerances less than 0.025 mm (0.001 in.) is not possible for conventional numerically controlled machines because the investment-cast blades aren't identical; the tip's spatial location can vary by as much as 0.75 mm (0.02 in.) from blade to blade. "So a unique tool path is required for each blade," explains Cliff Loucks, Sandia's project leader. "This is an ideal application for the adaptive machining techniques we've developed."

The new technique uses commercially available equipment controlled by Sandia-developed software. The blade is fixtured in a five-axis milling machine where a laser-based, structured-light sensor mounted on a tool holder collects three-dimensional data at several heights around the surface of the oversprayed blade. The data are analyzed to produce a CAD model that, because it is unique to the blade being processed, can specify the exact path for the grinding tool to follow as it travels around each individual blade. The overspray is removed in a single-pass, creep-feed grinding operation.

Sandia demonstrated the process last October in its Adaptive Machining Lab, and Pratt & Whitney expects to begin using the system in its production facility by the end of 1994.

Reprinted with permission from "Manufacturing Technology," A Sandia Technology Bulletin, August 1993.

Corrosion Control—The Only Way to Save the Infrastructure

The question is being asked "Why is the infrastructure falling down?" Why are the nation's roads, bridges, mass transportation systems, harbors, airports, waterways, water supply, waste and wastewater facilities so outdated, inadequate, or poorly maintained?

The U.S. infrastructure is valued at about $1.4 trillion, according to a recent study by the Office of Technology Assessment (OTA). The infrastructure's poor condition is attributed to three factors:

Factor 1: Most of the basic infrastructure is more than 20 years old and needs either major rehabilitation or replacement;

Factor 2: Shifts in population and transportation patterns have overburdened the infrastructure in major urban areas; and

Factor 3: Federal, state, and local governments face major budget problems.

Implied in each of these factors is the widespread practice of ignoring the necessity to design, operate, and maintain equipment and facilities for the longest service life possible. If infrastructure facilities and equipment are constructed of high quality materials using the current technologies and then properly maintained, the highest return on infrastructure investment can be expected and the longest possible service life obtained. The OTA report cited calculations by the Army Corps of Engineers indicating that maintaining and rehabilitating locks and dams by regular maintenance and structural repair have effectively doubled the lifetimes of these large structures. The Corps stated, "Barring a catastrophic event, these structures could last forever with good maintenance."

Cost Effectiveness Achievable Through Corrosion Control

Those industries using the existing corrosion prevention and control technologies have extended the service life of facilities and greatly reduced their costs. The two most commonly used materials of construction in the infrastructure are steel and concrete, both of which can be affected severely by corrosion. If attention is given to corrosion in the design, materials selection, construction, and then the operation and maintenance of facilities, even with the small additional up-front costs, millions of dollars in repair, maintenance, and replacement costs can be saved. The use of cathodic protection and protective coatings alone can prolong the service life of public works structures by many years.

Problems to Be Confronted

One government study indicated the annual losses to corrosion in the United States to be about four percent of the gross national product—about $230 billion (adjusted to 1992 dollars). The study, which was conducted by the National Bureau of Standards (now the National Institute of Science and Technology), estimated that 15% of that annual corrosion loss could be saved if existing corrosion control technologies were used.

Improved durability, resistance to operating stress, and protection against premature corrosion and failure can be achieved if existing materials, corrosion prevention, and corrosion control technologies were used. The OTA report stated that many of these new materials and corrosion control techniques, although tested and proven in field experience, are still not in widespread use. Industry is slow to adopt new materials and methods. Design engineers can be reluctant to use materials and techniques with which they are not familiar. Another reason for slow usage of the technology is that federal and state public policy decisions have not been made that would take advantage of the new technologies' cost effectiveness.

Another problem, according to the OTA report, is that most university civil engineering departments (the training grounds for many public works managers) do not teach courses in nondestructive evaluation or maintenance management. Few civil engineers have been introduced to the importance of corrosion prevention and control in their design and maintenance considerations.

One of the primary problems confronting the United States is summarized in this statement from the OTA report: "Corrosion has plagued all elements of the infrastructure for decades, and many technologies exist to combat this problem at both the design and maintenance levels. However, convincing state and local officials to invest limited funds in preventive methods is difficult."

Recommendations

A concerted effort is necessary to make design engineers and other decision makers at all levels aware of the cost effectiveness of using existing technology for corrosion prevention and control.

Engineers involved in design, maintenance, and rehabilitation of the infrastructure should be given the basic education necessary to use corrosion prevention and control technology in their work.

Efforts should be made to encourage the inclusion of corrosion design considerations and appropriate maintenance costs into the budgetary funding of infrastructure projects at all levels of government so that optimum service life can be achieved for all structural elements of the infrastructure.

NACE International, a professional society dedicated to the prevention and control of corrosion on all materials, has ongoing efforts to provide the technologies needed to reduce infrastructure losses due to corrosion. NACE believes that the use of existing corrosion control technologies can extend the service life of many existing facilities and can provide long service life to new facilities if proper design, operation, and maintenance procedures are used.

Thermal Spray Awards—NTSC'93

Dr. Mark Smith of Sandia (NM), the Chairman of the Thermal Spray Division of ASM International, announced awards for the NTSC'93 meeting during the Anaheim (CA) meeting in June of this year. The Awards Committee of the TSD gathered 40 panelists from the North American thermal spray community to judge the written submissions that were accepted for publication in the proceedings. The judges were asked to rank a group of papers according to the following guidelines.

1. Is the presentation style suitable? Is the paper clearly written (grammar and format), are the figures and tables well-presented; is the referencing correct? Is there a clear statement of objectives; and does the paper fulfill these? "Over-commercialism" of company names and products should be regarded as undesirable.
2. What is your assessment of the scientific and engineering merit of the work? Are the experiments well-thought out; is the discussion detailed; are the conclusions correct and detailed? Are the experiments logical; are they complete?
3. Is the work original? Is this first time you have seen the reporting of the work; is this an original interpretation of data; do the results and discussion represent new ideas?

Each paper was assessed by at least three of the panelists and their results averaged. The 11 papers listed below were judged as being of very high quality and received certificate awards from ASM International.

Influence of the Velocity of Plasma Sprayed Particles on the Splat Formation: S. Fantassi, M. Vardelle, A. Vardelle, and P. Fauchais, University of Limoges, France.

Characterization of Wear Damage in Coatings by Optical Profilometry: S. Dallaire, M. Dufour, and B. Gauthier, National Research Council Canada, Canada.

Mechanical Properties and Microstructure of Vacuum Plasma Sprayed NARLoy-Z: P.S. Chen, J.H. Sanders, and F.R. Zimmerman, Marshall Space Flight Center, USA, and Y.K. Liaw and T.N. McKechnie, Rockwell International, USA.

An Evaluation of Plasma Sprayed Tungsten for Fusion Reactors: R.A. Neiser and R.D. Watson, Sandia National Laboratories, USA, and G.R. Smolik, R.A. Anderl, and G.R. Longhurst, EG&G Idaho Inc., USA.

Control Modeling and Regulation of Thermal Spray Process: S.V. Kankanala, T. Demeny, and R.Kashani, Michigan Technological University, USA.

On the Gas Dynamics of HVOF Thermal Sprays: S.M. Settles, Penn State University, USA.

Deleading Paint by Vitrification Using Thermal Spray Technology: J.P. Petreanu and A. Kumar, US Army Corps of Engineers, USA.

Synthesis of Composite Materials by Reactive Plasma Spray Processing: R.W. Smith, Drexel University, USA, and E. Lugscheider, Aachen University of Technology, Germany.

Plasma Sprayed Powders and Coatings of Al-Li and Ti-Al Alloy Composites Prepared by a Plasma Spray Atomization Technique: K.A. Khor, F.Y.C. Boey, and M.J.Tan, Nanyang Tech. University, Singapore, and T. Sano, AIST, Japan.

Thermal Expansion of Metallic and Cermet Coatings: J. Ilavsky, C.C. Berndt, and H. Herman, SUNY at Stony Brook, USA, and M.B. Beardsley, Caterpillar Inc., USA.

Anode Arc Attachment Control Using Boundary-Layer Bleed Holes: S. Russ, E. Pfender, and J. Heberlein, University of Minnesota, USA.

Thermal Spray Awards for Volume 1 of JTST

The Editorial Committee and International Board of Review of the Journal of Thermal Spray Technology approved the formation of an Awards Committee under the Chairmanship of Dr. David Houck (Osram Sylvania Inc. (PA)). This Committee forwarded all the peer-reviewed papers of Volume 1 to five reviewers. The prime guideline for the judges was "to assess each paper on its own merit and with regard to the international standing of the thermal spray field."

The JTST Awards Committee stated that the purpose of making awards was to encourage and nurture excellence in the field of thermal spray research and engineering by recognizing JTST publications of exceptional quality. It is expected that such recognition will not only benefit the individual awardees, but also maintain the high-quality contributions that are being submitted to JTST.

The first two papers in the list below were awarded Plaques of Excellence, while the remaining were awarded Certificates of Merit. These awards were presented at NTSC'93 in Anaheim (CA).

Structure/Property Relationships of Sintered and Thermally Sprayed WC-Co: S.F. Wayne, GTE Laboratories Inc., USA, and S. Sampath, Osram Sylvania Inc., USA.

Diagnostics of Thermal Spraying Plasma Jets: P. Fauchais, J.F. Coudert, M. Vardelle, A. Vardelle, and A. Denoirjean, Laboratoire de Ceramiques Nouvelles, University of Limoges, France.

Significance of Quenching Stress in the Cohesion and Adhesion of Thermally Sprayed Coatings: S. Kuroda, T. Fukushima, and H. Kitahara, National Research Institute for Metals, Japan.

Thermal Spray Shape Deposition: L.E. Weiss, F.B. Prinz, D.A. Adams, and D.P. Siewiorek, Carnegie Mellon University, USA.

Flattening and Solidification of Thermally Sprayed Particles: C. Moreau, P. Cielo, and M. Lamontagne, National Research Council of Canada, Canada.

Do you have literature or news you'd like highlighted in this feature? Send your contributions to the editor.

Fundamental Considerations and Design Selection of Water Cooling Systems—An Industrial Note

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Summary

Both plasma and HVOF processes need high water pressure to cool the electrodes and other surfaces exposed to the flame or arc. The high heat fluxes in the plasma, HVOF combustion chamber and barrel require precision water cooling. In addition, if water is not deionized and demineralized (for example by the use of distilled water), minerals will build up on the heat exchange surfaces of the torches due to nucleate boiling that occurs on the hottest areas of these devices. Any minerals that may be deposited act as an insulator and reduce the heat transfer—thereby further increasing the temperature of the torch parts and creating more intense nucleate boiling. Eventually, the wall may be hot enough to melt.

In summary, it is important that clean mineral-pure water be used to cool torch surfaces and that it be of a low enough initial temperature so that its temperature rise in the device, due to the heat input (25 to 40 °F [14 to 22 °C]), does not raise the output water temperature above a reasonable temperature level (140 °F [60 °C]).

The design of the water supply system is further complicated by the fact that different installation areas have varying maximum ambient temperatures. For example, in the southern United States, city or plant water temperatures might be as high as 100 °F (37.8 °C) on some days. This might raise the outlet temperature of the water in the torch above tolerable levels with an inadequately designed system. Secondary water quality used to cool the heat exchangers that supply the distilled water for torches and that is sometimes used to cool spray torches (but not recommended for reasons to be outlined) must be evaluated.

Sample Calculations

HVOF and plasma torches require dissipation of significant amounts of heat,; as much as 40% of the heat input. A simple calculation for the cooling water requirements of a HVOF system is presented for instructional purposes.

Assumptions:

Fuel input:	4 gpm (15.14 L/min)
Heat value of fuel:	134,000 Btu/gal (37.35 MJ/L)
Heat to be absorbed:	39% of input
Inlet supply water temperature:	70 °F (21 °C)
Maximum water temperature desirable:	130 °F (54 °C)

The combustion energy = 4 gpm × 134,000 Btu/gal × 60 = 536,000 Btu/h (565.5 MJ/h). The energy that needs to be absorbed by the water = 0.39 × 536,000 = 210,168 Btu/h (221.7 MJ/h). Therefore, the cooling water flow rate required can be calculated as follows:

$$Q = WCp\Delta T \qquad [1]$$

where Q is the heat dissipated (Btu/h), W is the weight of water (lb/h), Cp is the specific heat of water (Btu/lb °F), and ΔT is the temperature rise (°F). Therefore:

$$W = Q/(Cp\Delta T) = 3503 \text{ lb/h or } 7 \text{ gpm } (26.5 \text{ L/min}) \qquad [2]$$

From the above analysis, a water flow of 7 gpm (26.5 Lpm) is required to limit the temperature rise to 60 °F (33 °C), therefore limiting the maximum outlet water temperature to 130 °F (54 °C). The intention of this industrial note, after providing reasons for the requirement of heat dissipation, is to recommend and discuss various options for users with particular requirements.

Systems Available

Various water heat dissipation options are available depending on the specific situation, plant design, and location. The choices range from simply piping city, river or plant water through a water-to-water heat exchanger and pumping clean demineralized water through the torch at the proper pressure, to a totally enclosed, air-cooled refrigerated water recirculating system to dissipate energy in the normal refrigeration cycle fashion. Such [redacted] are already used widely to cool injection molding machines and vacuum furnaces. Thermal spray devices require higher than normal water pressure (10 atm minimum) to obtain adequate flow through the hoses and gun and to suppress nucleate boiling. Less boiling occurs at higher pressures and higher pressures allow for the higher pressure drops which ensure that a flow velocity of 20 ft/s (6.1 m/s) passes over the hot sections and therefore sweeps away any nucleate bubbles developed that limit heat transfer. There are three prime options; as follows.

1. *Water-to-Water Heat Exchangers*

In this mode, plant cooling water or city water are used on the primary side of a water-to-water exchanger to cool deionized water which is circulated through the spray torch (Fig. 1). An adequate bulk of water is provided so that small losses during hose disconnections do not trigger low water limit controls. In some models a flow side arm is built in to continually deionize a portion of the water and maintain the high level of purity without purchasing and adding water from time to time.

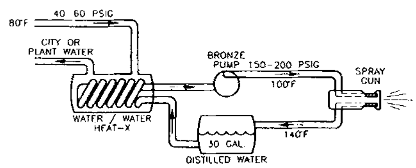

Fig. 1 Water-to-water heat exchanger.

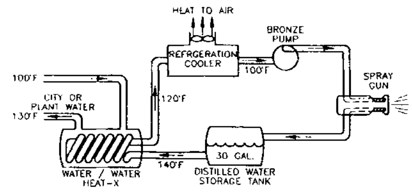

Fig. 2 Refrigerated system heat exchanger. The totally refrigerated system does not include the water-to-water heat exchanger.

2. *Partially Refrigerated Systems*

If either the cooling water supply availability to cool the heat exchanger is inadequate or the maximum temperature of the cooling water is too high, a second option is to utilize a booster refrigerated cooler. In this case, the plant water is used to absorb a major part of the cooling heat load and an in-line refrigerated cooling package absorbs the remaining energy to bring the cooling water temperature to a reasonable level (Fig. 2).

A typical example would be an installation in Texas where the summertime inland water cooling temperatures are 100 °F (37.8 °C). Under these circumstances, to supply water to adequately cool (see Eq ?) and still limit the output temperature to 140 °F (60 °C), the inlet temperature must be 80 °F (27 °C). Therefore, it is necessary to reduce by 20 °F (11 °C) the temperature of water flowing at 11 gpm (41.6 Lpm) through the torch. A heat extraction of 164,130 Btu/h (173.1 MJ/h) or 14 tons of refrigeration (each ton of refrigeration extracts 200 Btu/min [211 KJ/min]) is required.

The advantage of the partial refrigeration system is that it minimizes the size of the refrigeration unit required and thus minimizes capital costs. However, the use of city or plant water is an added expense that must be considered.

3. *Totally Refrigerated System*

An air-cooled refrigerated system is required where it is necessary to totally isolate the spray system from any plant water. Although this is often the most capital-intensive approach, it does provide total independence. In this system the ambient air cools the refrigerant condensers in the cooling unit and higher air temperatures will require a larger fan. A schematic of the system would resemble Fig. 2 without the city water heat exchanger.

Conclusion

Several options for cooling both plasma and HVOF thermal spray coating systems have been outlined. A specific design must be considered for each installation to minimize costs and match the cooling unit to the conditions and equipment in the plant. Each installation should be optimized with respect to performance and costs. The following information is required for such an analysis.

1. Type of water available (city, cooling tower, plant refrigerated).
2. Temperature of available water.
3. Maximum flow of water available.
4. Heat that can be absorbed in water available and maximum temperature to which it can be raised.
5. Maximum ambient air temperature that would be expected during continuous operation of the spray system.
6. If a refrigerated cooler is required, is it possible to locate the air-cooled condenser unit outside the building—is space available?
7. Should the unit include an automatic deionized water makeup or will deionized water be added as needed?

This industrial note has been edited by C.C. Berndt (SUNY at Stony Brook) from an industrial bulletin of Hobart/Tafa. The original bulletin was titled "Fundamentals/Design Selection Water Cooling Systems,"

NIST-Industry Workshop on Thermal Spray Coatings Research

Gaithersburg, MD, July 20, 1992

Reported by: S.J. Dapkunas, National Institute of Standards, and Technology, Gaithersburg, MD 20899-OMI

1. Introduction

In 1992, a comprehensive survey of current federal materials research programs and plans for new initiatives, known as the Advanced Materials and Processing Program (AMPP), was developed under the auspices of the Office of Science and Technology Policy. The goal of the AMPP is to improve the manufacture and performance of materials to enhance the nation's quality of life, security, industrial productivity, and economic-growth.[1] One element of the program addresses what have become known as "Functionally Gradient Materials" (FGM). This class of materials is distinguished by properties that vary with material thickness. These gradients in properties such as hardness, thermal conductivity, and chemical stability contribute to the improved performance of components. For example, the wear resistance of a soft but tough material can be greatly improved by the application of a harder but compliant surface deposit overlayed with a very hard but brittle material. The concept of gradations of material properties to optimize performance is not new, but increased awareness of the opportunities for improved control of properties and the ability to tailor microstructures continuously through a thickness has fostered a view of these materials as a distinctive class whose potential has not been fulfilled. Typically these materials' compositions vary from metallic to ceramic over a thickness of up to several millimeters. The unique approach now taken to graded materials in FGM research is to target specific properties at the extremes of the material's thickness, thermal conductivity, and thermal expansion, for example, and to tailor microstructure, porosity, or other features to meet those properties. Extensive research to achieve this capability through understanding of processing/microstructure/property relationships has been initiated through material synthesis techniques as varied as thermal spray, chemical vapor deposition, and self-propagating high-temperature synthesis.[2]

Thermal spraying of coatings, due to high material deposition rates and relatively low capital cost, has become a primary industrial method of synthesizing materials with varying composition and microstructure. The North American thermal spray market was over $600 million in 1990 and is projected to reach $2 billion per year by the year 2000, a growth rate of 7 to 8% per year. The largest portions of this market are in powder consumables and coating services. The growth projected is largely based on the increased use of ceramic coatings for thermal barriers and clearance control on aircraft gas turbines, some of which have close to 5500 parts which are thermal spray coated.[3] Similarly, the market for ceramic powders used in thermal spraying is a significant portion of the advanced ceramic powders market and is expected to experience a growth rate of 4% per year through 1995[4] with annual consumption of oxides and carbides reaching over 2 million kilograms by the turn of the century.[5]

The thermal spray industry is diverse. In addition to the aircraft engine applications noted above, thermal spray deposited coatings are applied to fossil-fueled boilers and chemical processing vessels to control corrosion, to automotive bodies and mechanical components for cosmetic and wear reduction purposes, to electrical components for insulation, and research is under way to increase efficiency of reciprocating engines through application of thermal barrier coatings to piston crowns. Thermal spray processes are also used to refurbish worn mechanical components, thereby reducing scrap and replacement costs.[6] The companies which supply materials and services for these applications are likewise diverse in size and capabilities. The 1992 Thermal Spray Buyers Guide lists 38 powder suppliers, 25 equipment suppliers, and 40 contract applicators,[7] in addition to the major automotive and aerospace companies that are the large single-site users of the technology.

Recognition of the size of this industry, the varied interests and skills of the scientists and engineers involved, and the changes in the field have fostered the growth of technical and trade organizations to serve the field's technical communication needs. The primary domestic technical society addressing the community is the Thermal Spray Division of ASM International, which was formed in 1987 and now sponsors the publication of the Journal of Thermal Spray Technology. Trade and marketing issues are served by the International Thermal Spray Association. Although research results are available in a wide venue of scientific and engineering journals, the most comprehensive summaries of current research and technical developments are found in the proceedings of the 13 International Thermal Spray Conferences (1956-1992) and the 4 National Thermal Spray Conferences (1981-1991). These conferences now attract over 1000 attendees each.

2. Thermal Spray Processing

Thermal spray coatings are applied by injecting the material to be deposited into a high-velocity hot gas directed to the substrate of interest. The coating feedstock is generally a powder, but wires and rods are also widely used and the controlled feeding of this material allows the development of a coating whose composition and microstructure varies with thickness. The high-temperature gas is obtained either by the development of a plasma generated by passing an inert gas through a set of high voltage electrodes or by combustion of reactive gases in the torch itself. Plasma spraying and flame or high velocity oxygen fuel (HVOF) are the general descriptions of these processes respectively. Process conditions can vary widely and have significant influence on the microstructure and properties of the deposited material. Table 1, taken from Ref 8, identifies some of the pertinent features of the various thermal spray processes. The plasma spray process has been adapted for operation in vacuum to increase the density of the deposit and in inert gas filled chambers to prevent oxidation of the material sprayed.

The high temperatures and velocities of the thermal spray processes make measurement and control of process parameters difficult. Therefore, although widely used, production of quality coatings largely depends on the experience and intuition of skilled equipment operators.

Microstructures of deposited coatings are complex, particularly for graded structures. The extreme thermal conditions to which feedstocks are subjected, high cooling or solidification rates, reaction during transit to the workpiece (substrate), and morphological features resulting from high but variable impact velocities combine to make prediction and control of microstructures difficult. Hence, much of the material produced commercially is the result of empirical studies relating gross process parameters to microstructures and to performance in actual application. Typical thermal sprayed coatings exhibit overlapping lamella resulting from successive impacting particles of molten or very plastic material, oxide films surrounding the lamella, irregularly distributed porosity, rough interfaces between layers of different composition, and cracks resulting from shrinkage during cooling. These features make quantitative analysis and specification difficult.

Properties of thermal sprayed coatings are difficult to measure, especially in service. Tensile strength, elastic modulus, thermal conductivity, and fracture toughness are important coating properties but the most sought after property is adhesion to the substrate. Typically, this is determined by the tensile adhesion test, ASTM C633-79. Large variations in adhesion strength measured by C633-79 have been shown to be typical.[9] Other properties related to performance, such as erosion or corrosion resistance, are routinely measured and related to operating conditions in specific applications.

3. Workshop Objective and Structure

The objectives of the workshop were to identify (1) the research required to improve processing reproducibility and performance prediction, (2) opportunities for collaboration between NIST and industrial researchers, and (3) mechanisms of effective dissemination of research results to the thermal spray community. This approach is more focused than some earlier studies, Ref 10 and 11 for example, which include thermal spray in general assessments of coating research needs and do not

Table 1 Characteristics of Thermal Spray Deposition of Tungsten Carbide-Cobalt Coatings

	HVOF	Standard plasma	High-velocity plasma
Flame temperature, °C	2,760	11,100	11,100
Gas velocity	Mach 4	Subsonic	Mach 1
Hardness, (DPH 300)	1,050	750	950
Porosity, %	0	<2	<1
Typical bond strength, MPa	69	55	69
Thickness limit, mm	1.52	0.76	0.38

specifically address industrial processing concerns.

Invited attendees represented a broad spectrum of the thermal spray industry including powder suppliers, equipment manufacturers, and applicators and users of thermal spray coatings. In addition, researchers from academia and federal laboratories with active programs in thermal spray, as well as representatives of the principal organizations through which the thermal spray community communicates were invited.

The workshop was structured to present visitors with an overview of unique NIST analytical and materials characterization techniques that are viewed as providing improved capabilities to understand the role of processing on performance and properties. Members of the Materials Science and Engineering Laboratory and the Chemical Science and Technology Laboratory staffs reviewed chemical and compositional mapping of microstructures, thermal properties measurements, and powder characterization techniques developed and utilized at NIST.

A crucial aspect of the workshop was to solicit the view of industry on their requirements for measurement-related research. To accomplish this, industrial and academic representatives described the general requirements for measurement of process parameters, mechanical properties, microstructural analysis, and modeling of the thermal spray process. The specific issues of the automotive industry were addressed by representatives from Ford and General Motors, who emphasized performance prediction.

To facilitate implementation of research results, NIST personnel described the various mechanisms, such as Cooperative Research and Development Agreements and the Advanced Technology Program, through which collaborative research can be conducted. Similarly, the past chairman of the ASM International Thermal Spray Division described that organization's structure and means of coordinating dissemination of information.

Following these general presentations, working groups convened to determine specific research topics that are of importance to the thermal spray industry. These groups addressed process measurement and control with an emphasis on powder characterization, coating evaluation, performance evaluation, and process modeling.

4. Research Issues

The following research issues and needs were determined by consensus through the working group discussions.

4.1 *Processing Measurement and Control*

Powders are the predominant form of thermal spray feedstock and are increasingly recognized as having a strong influence on the microstructure and performance of coatings. Therefore, consideration of powder characteristics as a process variable is warranted. Powders for thermal spray deposition are synthesized by several techniques and are generally specified by bulk composition and particle size. Metallic powders are usually formed by atomization from an alloy melt while oxide, carbide, and other ceramic compositions are formed by crushing and grinding larger material to the desired size or by spray drying fine powder with an organic binder to obtain the size required. The type of ceramic processing used influences properties such as powder shape, phase content, friability, and flowability.

The powder characterization working group aimed to identify those powders that were of greatest interest to industry and to determine which powder properties were most critical to spray process control and which types of standardized testing are required or need improved technique.

Powders can be divided into those that are intended for coating use at elevated temperatures and those that are exposed to ambient temperatures. Among the former are the zirconia-containing thermal barrier and M-CrAlY (nickel, cobalt, and iron as the primary constituent(s) with chromium, aluminum, and yttrium alloying additions) coatings applied to gas turbine components, and among the latter are tungsten carbide and aluminum oxide utilized for wear protection. For high-temperature applications, 7 to 8% yttria-stabilized zirconia was found to be of greatest interest. For this powder in particular, synthesis technique has a pronounced effect on powder shape, porosity and other features that affect both spraying and deposit formation. These synthesis-related features were felt to be the cause of variations in measurements required for powder specifi- cation. Specific working group recommendations for research to resolve these issues are as follows:

- Calibration and cross correlation of powder size measurements by powder producers and users should be conducted using well-characterized reference lots of material as has been done in the fine ceramics industry. This would be most effectively conducted through the distribution of captive cells of material. This research would have immediate benefits to the thermal spray industry.
- A standard should be written for the size analysis of powders in the 10 μm size range including sample preparation technique. Major interest focused on the analysis of gas-atomized and spray-dried powder that should be the primary materials studied. Size analysis of nonspherical powders is not currently conducted in spite of the fact that significant amounts of this material are used. Optical size measurement methods are desired, particularly by users.
- Techniques for measurement of specific surface area, phase composition, and chemical composition require development for thermal spray powders. In particular, analysis techniques for impurities such as silica, alumina, sodium, hafnium, uranium, and thorium in zirconia are needed. Apparent density measurement techniques for spray-dried powders require development to allow improved process control.
- The ultimate test of a powder is its behavior in the thermal spray process and the working group determined that development of a standardized spray test that would determine deposition efficiency is warranted. It was the group consensus that an impartial institution such as NIST would be extended cooperation from the thermal spray community to develop such testing procedures.
- Sensors to measure thermal spray process conditions were clearly identified as requiring development. Conditions that require measurement include particle temperature, particle velocity, in-process coating density, and deposit thickness. It was felt that an emphasis should be placed on high-velocity spraying techniques.

Significantly, powder producers expressed willingness to contribute to the development of the research recommended through contribution of materials, analysis procedures, and the conduct of analyses as part of a collaborative research effort.

4.2 *Coating Evaluation*

Measurement of coating properties and analysis of coating microstructure and microchemistry are critical to both the evaluation of thermal spray processes as well as the prediction of performance. Working group members identified this broad range of immediately useful research which would enhance productivity and effective application.

- The inhomogeneous microstructure typically produced is difficult to analyze due to the presence of constituents as diverse as gross porosity, interlamellar phases, metallic glasses, and oxides. The situation is made more complex by the presence of metastable phases resulting from rapid solidification of powders upon impact with the substrate. Development of reproducible methods to provide quantitative analysis of both gross and subtle features by optical and electron microscopy are needed. It was suggested that a microstructural atlas of coatings would be of use to industry.
- Development of techniques of x-ray diffraction that can routinely be utilized to determine phase content and composition presents challenges that if overcome could provide improved understanding of the role of processing conditions. Similarly, techniques to quantitatively assess phase fraction, residual stress, grain size, and solute levels requires development. The identification of metastable phases that have weak x-ray diffraction patterns is confounded by accompanying fine grain sizes, internal stresses, solute gradients, and texturing effects. This analysis is not performed, although the presence of these features can have a significant effect on performance. Characterization of microcracking, which can have significant effects on strength, fracture toughness, thermal conductivity, and corrosion protection, is not easily or well conducted. Development of methods to analyze microcracking can also provide insight into the mechanisms of coating failure.
- Measurement techniques to determine the mechanical properties of thermal sprayed coatings are not well defined although research addressing this topic has been conducted.[12-13] Adhesion to the substrate, cohesion within a coating, and properties of the coating material, particularly when graded, are important to coating design and understanding of the role of processing parameters. Typically adhesion is measured by use of the tensile adhesion test (TAT, ASTM-C633-79) originally developed for evaluation of zinc coatings on steel. This test, which consists of pulling the coating from the substrate by means of a tab epoxied to the coating, is limited to the strength of the epoxy. It is not conducted above 200 °C and provides only rough quality control guidance in contrast to more elegant techniques applied to homogeneous thin films.[14] A test methodology that can be readily conducted by applicators and provides an understanding of mode of failure, failure initiation site, strain to failure, and other pertinent data for the coating-substrate system is desired. Data on the properties of coating materials is either gathered from handbook values of bulk material of similar composition or measured on coatings removed from a substrate. These data are either not representative of the coating or neglect the role of interfacial constraint at the substrate.
- Thermal properties are particularly important for graded, insulating coatings. It was noted that developing both an ability to measure thermal conductivity and to model this property based on microstructural parameters would enhance industry's ability to design coatings for particular applications.
- Group participants suggested that round-robin programs to establish a basis for comparison of test methodologies would be productive as has been shown in a recent exercise to evaluate techniques of metallographic preparation of tungsten carbide coatings. This latter effort, which entailed the distribution and analysis of 1800 samples, has provided evidence of the value of standard reference materials and standard evaluation techniques. In the long-term, databases on thermal, mechanical, and other properties may be feasibly developed by industry, if accepted measurement and analysis techniques are available.

4.3 *Performance Evaluation*

Performance evaluation and prediction techniques are vital to the competitiveness of material producers and coating vendors and hence are usually closely held. The performance evaluation group identified several general service-related issues that should guide the development of a research agenda.

- Although research on current applications is of value, attention should be directed to emerging applications with major growth potential. These applications include thermal barrier coatings for nonaircraft engine applications in the automotive industry, corrosion-resistant coatings of value to the chemical industry, and electrical insulators applied to electromechanical equipment used in various commercial products.
- Tungsten carbide/cobalt wear-resistant coatings are a large thermal spray market. Less costly alternatives to this material are of interest, and research to assess their performance limits would be of benefit.
- Corrosion-resistant coatings for aqueous and other environments are of interest to several industries. An improved understanding of mechanisms of deterioration that would allow better material selection and performance prediction is desired. In terms of characterization techniques, the ability to measure permeability of coatings on a substrate was specifically cited.
- Measures of performance are application-specific, and the occurrence of unforeseen, unmeasured operating conditions limits predictive capabilities. In this context, it was emphasized that understanding mechanisms of coating failure would be of value, particularly in relating laboratory assessments to field measurements.
- The ability to determine the condition and predict the remaining life of coatings is valuable. This capability and the desire to inspect coatings, without reliance on test coupons included in production lines, led to the recommendation of development of in situ nondestructive evaluation techniques.

In the extreme, industrial representatives stated the desire to be able to specify performance based solely on processing conditions. This is recognized as a long-term goal that requires significant understanding of the particular mechanisms of deterioration likely for an application and the role of coating properties and microstructure in that mechanism.

4.4 *Process Modeling*

Process modeling was recognized as the activity that binds several aspects of thermal spray coating together. Modeling of the process from the torch to the coating deposition was cited as necessary for process design, control, and automation. Group recommendations for research included the following specific items.

- Most modeling research has been directed to plasma spraying. The increased interest in high-velocity oxygen-fueled spraying argues for the development of models of this process wherein higher velocities and deposition rates present challenges.
- For all processes, models of the development or evolution of the complex microstructure are needed. Microstructural development models would provide a link between processing and properties with the potential for better property control and consistency.
- Microstructural development models should include understanding of the nature of impaction and coalescence of droplets, the formation of defects, and the fine features of bonding to the substrate.
- Modeling of thermal spray torch parameters is important for process improvement. Specifically, models of the thermal and flow behavior of the hot gases emanating from the torch and the behavior of particles in flight to the workpiece were cited as necessary.

Significantly, it was stated that the process modeling efforts should be integrated with both diagnostic developments and process design and that the specific classes of material addressed should be recommended by industry.

5. Conclusions

The workshop was successful in identifying many of the key problem areas in thermal spray coating technology. A broad spectrum of issues in this complex process that reflected the concerns of different industries was addressed in the discussion groups. The active participation of the attendees reflects the interest industry has in the development of a research agenda which addresses improved process reproducibility and performance prediction. It is significant to note that most attendees expressed a willingness both to identify important issues that limit the technology and to participate in collaborative research projects to which they would contribute materials, services, and expertise. A key to this willingness was the realization that industrial and academic capabilities in material processing could be effectively utilized in conjunction with NIST's measurement, modeling, and characterization capabilities. Opportunities for transfer of research results to industry through established thermal spray technical organizations were clarified. As a result of the workshop, NIST will synthesize consensus project plans for the consideration of the attendees and initiate collaborative research where sufficient interest warrants.

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