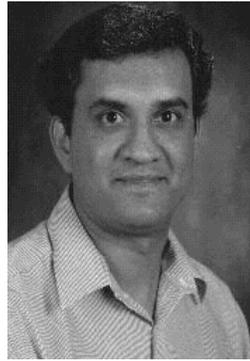


NSF Materials Research
Science and Engineering Center

CENTER FOR THERMAL SPRAY RESEARCH

Going Beyond The Surface

Message from the Director: Dr. Sanjay Sampath



It gives me great pleasure to introduce our third annual newsletter at a time when the Center's activity is at an all time high and our research can be described in one word "thriving". A number of exciting things have happened over the last year and this newsletter highlights some of the activities and reflects on our achievements.

The Center went through a major site review from NSF in September 2003. This usually conducted once in three years and involves several outside peers along with NSF staff members. I am pleased to report that our review was highly successful. This is largely due to the dedicated efforts and contributions by the Center participants and particularly our post-docs and students. Especially noteworthy were the introduction of new ideas and concepts by our interdisciplinary participants. The successful site visit allows for the continuation of Center support for another two years and also sets the stage for the re-competition that begins in September 2004.

The Center saw other important transitions during the year. Prof. Herb Herman formally retired and transitioned to the status of Professor Emeritus. In his new role Herb will continue to mentor students and participate in re-

search discussions. Last year also saw the arrival of our newest faculty member: Prof. Andrew Gouldstone. Andrew joined the Center and the Materials Science Department as an assistant professor in the fall of 2003. Andrew graduated with Ph.D. from MIT and followed that with 2 year post-doc at Harvard School of Public Health. Andrew is a breath of fresh air. Although he had no prior experience in thermal spray, he has been a quick learner and has already made significant contributions to our understanding of multi-scale mechanical properties of thermal sprayed coatings. Andrew has become the "darling" to the students. He has an infectious enthusiasm for learning, teaching and inspiring students and he is leaving a mark on them. Andrew will be a major *tour-de-force* in the field of thermal spray and materials science in the coming years.

The Center is making breakthrough strides in understanding the process-materials interactions. Aided by integrated diagnostics and modeling, our post-docs have made extraordinary progress in understanding process sensitivity and process variability with a particular emphasis on TBCs. The basic research knowledge developed through the NSF research has already paid important dividends to industry. The newsletter highlights some of these activities.

Our industrial consortium to promote knowledge transfer

was successfully renewed by all members for a second year despite trying economic times. The Consortium is now a well oiled machine and has been a very productive entity in developing interactions between the Center and the various industrial partners. Lysa discusses the activities of the consortium in this newsletter and I urge you to consider participation.

This year under Lysa's leadership we are embarking on a new strategy to significantly expand and enhance our educational outreach to the K-12 sector. Through an NSF research experience for teachers program, we are planning a two tiered outreach effort to introduce concepts of materials science, surface engineering and thermal spray to schools. Lysa describes this pilot program in this newsletter. We believe this is an important venture for the future of our technology encourage you to participate in this endeavor.

Finally, we are actively preparing the program for summer workshop 2004 (July 14-16). This year the theme will be "Materials and Processes for Energy Systems" and will honor Prof. Herman on the occasion of his 70th Birthday. The workshop features eminent speakers from industry and academia and will also conclude with an alumni reunion event. We are also hoping that we will have occupancy of our new building which is coming on line within the next couple of months. We hope to see you this summer at Stony Brook and help us to celebrate our many achievements and set the stage for the future!

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Special points of interest:

- ??Center founder, Herb Herman Retires
- ??Tech Highlights: Precursor Plasma Spraying
- ??Indentation: A Simple Tool with Powerful Capabilities
- ??Comprehensive Look at TBC Structures Leads to Improved High Temperature Performance
- ??Alumni Highlights: Dr. S. Rangaswamy

Center Founder, Herb Herman, Retires from Teaching

After a 35 year career that started with his Ph.D in Metallurgy and Materials Science at Northwestern University and a Fulbright Scholarship at the University of Paris, Distinguished Professor Herbert Herman has retired from his teaching responsibilities at the State University of New York at Stony Brook.

Herb's career in thermal spray started back in the mid 1970's when as a young professor, one of his graduate student's "stumbled" upon a novel deposition technology for non-equilibrium materials which turned out to be thermal spray. Fascinated by the science involved in the process, Herb continued his research into the technology for the next 30 years and quickly became one of the most vocal and dedicated researchers in the field.

Herb played a pioneering role in bringing science into the industrial technology of thermal spray and intro-

duced a scientific approach that transformed the field. In addition, Herb played a key role in forming the Thermal Spray Division of ASM International and was the Division's founding Chairman. For his significant contributions to the industry, Herb was elected into the Thermal Spray Hall of Fame.

Herb's charismatic presence, both in and out of the classroom, energized everyone from students to thermal spray practitioners and created a following of believers in the technology. Many of today's leading scientists and industrial researchers in TS chose to pursue this industry as a direct result of Herb's own motivation and love of the process. Students from around the world found Herb to be more than just a mentor but a surrogate father and their gratitude and devotion towards him is clearly evident.

In addition to his role in the thermal spray community, Herb also played a

significant role in the materials science and technology community by serving as editor for a host of technical journals including Treatise on Materials Science & Technology, Materials Science and Engineering (A) and Ocean Technology Series. Herb also has fellowship status in two materials societies including ASM International and the American Ceramic Society.



Some of Herb's past students gather at a dinner honoring his career and achievements.

Consortium on Thermal Spray Technology: Howmet Joins As Newest Member

Since the spring of 2002, the *Center for Thermal Spray Research* has hosted a very successful collaborative research consortium consisting now of eleven leading industrial users and manufacturers of thermal spray equipment and materials. Through their combined resources and the facilities and personnel of the *Center*, industrially relevant, pre-competitive research agendas are established and carried out. This year, the Consortium is proud to welcome its newest member Howmet Castings, a leading manufacturer of components for jet aircraft, gas turbines and other advanced technology industries.

This year's research builds upon the YSZ powder morphology studies from the previous year's work to develop process maps for coating systems. The maps that are generated come from the use of highly sophisticated visualization and diagnostic equipment which create acceptable process windows for industrial coatings. The data that is being generated now will allow users to intelligently select parameters and predict the outcome

(performance) of the coating in the very near future.

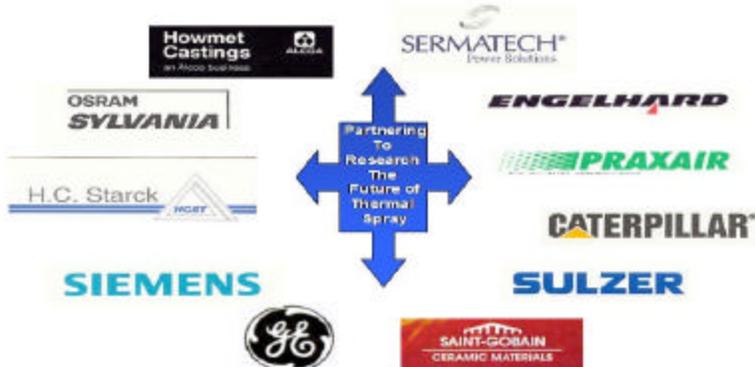
In year one, the research knowledge transfer effort focused on addressing and understanding:

- ?? YSZ powder morphologies and interactions/behaviour within the plasma
- ?? Sintering behaviour of YSZ coatings

- ?? MCrAlY HVOF, APS and VPS bond coat elastic/plastic properties.

Consortium members meet two to three times per year for research updates and networking opportunities. In addition, members receive relevant publications from the *Center* throughout the course of the year.

For more information on membership, please contact Lysa Russo at: lysa.russo@stonybrook.edu



Research Experiences for Teachers: New Program for CTSR

A critically important aspect of the CTSR is its commitment to educational and industrial outreach to the community at large. As such, the CTSR facilitates summer research experiences for middle school and high school teachers of science, technology and math. The Research Experiences for Teachers (RET) is a five week program that utilizes hands-on laboratory experiences to familiarize teachers with the fundamentals of scientific research and some of the latest advances being made in materials science and engineering.

All middle and high school math and science teachers who hold a New York State Teacher's Certificate are eligible to the program. Physics, chemistry and math teachers as well as teachers in high minority, under-represented school districts are strongly encouraged to apply.

The program runs for five weeks, starting July 6th and ending the week of August 6th and is held at the Center for Thermal Spray Research located on the SUNY Stony Brook campus. Teachers each receive a stipend of \$4,000 for completing the program.

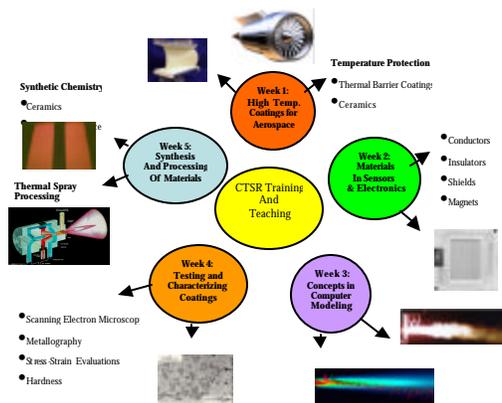
During the course of the program, teachers will:

- ?? Participate in research projects using CTSR equipment and facilities

- ?? Gather insight into laboratory design
- ?? Develop new and exciting curriculum for their classroom
- ?? Learn how to utilize sophisticated laboratory equipment.

Each week, a different research theme is explored. They include:

- ?? High Temperature Coatings for Aerospace Applications
- ?? Materials in Sensors and Electronics
- ?? Concepts in Computer Modeling
- ?? Testing and Characterization of Coatings



"Now I understand how IMPORTANT it truly is for us to become knowledgeable on the subject of sciences. The enthusiasm we possess in the classroom DOES play a large role on what the children take a like to", Jaclyn Mazzurco, LI elementary school teacher.

- ?? Synthesis and Processing of Materials

At the end of each week, teachers will have the opportunity to network and develop a detailed curriculum project on that week's topic. The teaching module that is developed by the group can be used by each of the teachers, in their own classrooms, as well as be posted on the Center's website so it can be shared by other interested teachers.

This program is designed to give teachers an up-close, personal look at scientific work on the research level and have the opportunity of working with leading faculty and scientist in one of the country's finest research universities.

If, interested please contact Lysa Russo at lysa.russo@stonybrook.edu to obtain additional information on the RET program as well as obtain copies of the application forms.

High School Students Spend Semester Studying Thermal Spray

This past semester, the CTSR was delighted to offer research internship opportunities to two very talented Syosset High School sophomores. On weekends starting in November 2003 through March 2004, these young ladies would come to the laboratory to experience, first hand exactly what is involved in the undertaking of a scientific program.

Their selected project involved the metallographic preparation and evaluation of Ni-Al bond coatings deposited using four commercially viable processes: high velocity oxygen fuel (HVOF), twin wire electric arc, vacuum plasma spray and air plasma spray. During their studies, they learned specifics about each

process and how coating microstructures can be varied significantly depending upon the process selected.



Examining Sprayed Coatings Using Optical Microscopy.

To evaluate coating microstructures, both optical and scanning electron microscopy was employed. The nature of splats, oxidation and pores were fully explored and understood for each coating structure.

As part of the project, a final report was written summarizing the spray processes that were selected, polishing procedures and evaluation techniques used to examine the sprayed coatings and what the findings were for percent oxidation and porosity.

Programs like these provide gateways for young people to explore the many opportunities available in the world of science and engineering!

Tech Highlight: Single Step Synthesis and Deposition of Functional Oxides by Precursor Plasma Spray

A single step approach to make functional oxide ceramic coatings by radio frequency (RF) plasma spraying technique (PPS) starting from solution precursors is under investigation. This emerging method of thermal spraying, utilizes molecularly mixed precursor liquids, which basically avoids the handling and selection of powders, enabling new avenues for developing complex functional oxide coatings. PPS has been successfully employed to deposit thin/thick films of spherical and nanostructured deposits of yttrium aluminum garnet (YAG), yttrium iron garnet (YIG), lanthanum strontium manganate (LSM) and yttrium zirconium titanates. Red emission from highly crystalline and luminescent films of europium-doped YAG and yttrium oxide (Eu-Y₂O₃) produced by this method are shown in Fig.1.

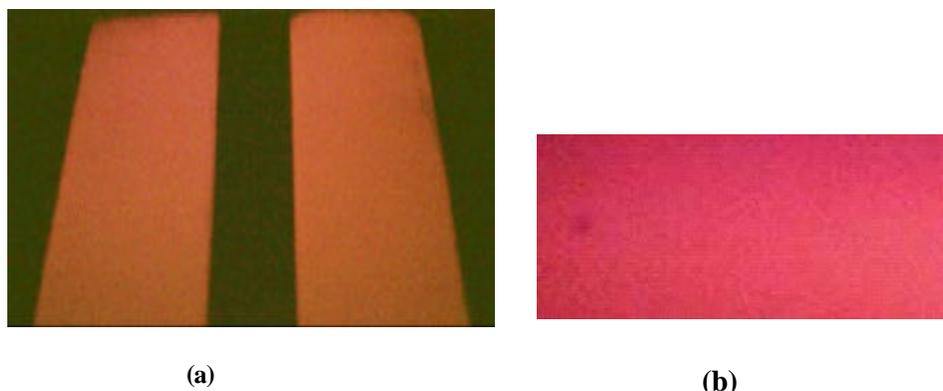


Fig. 1: Red emission from (a) Eu-YAG and (b) Eu-Y₂O₃ films when excited with a Mineralight UV lamp.

simplicity of the process. Formation of randomly oriented coatings by the present approach, confirms a homogeneous nucleation process. Generally, textured coatings are formed during the chemical vapor deposition processes indicating a heterogeneous nucleation. Free from electrode contamination of the products, axial injection, larger hot zone, controllable temperature are the key advantages.

Precursor Plasma Spraying

This process may be economically viable to develop large area coatings of luminescent phosphors as well as other ceramic coatings for magnetic and fuel cell applications. CTSR's interdisciplinary scientists in collaboration with Prof. Levi's group at University of California, Santa Barbara also investigating the non-equilibrium phase evolution during plasma spraying of multi-component oxides from inorganic precursors. By proper feedstock design and control, and deposition optimization, we are also exploring the composition and microstructural space in a discrete combinatorial manner and access far-from equilibrium configurations that offer practical opportunities and intriguing scientific issues.

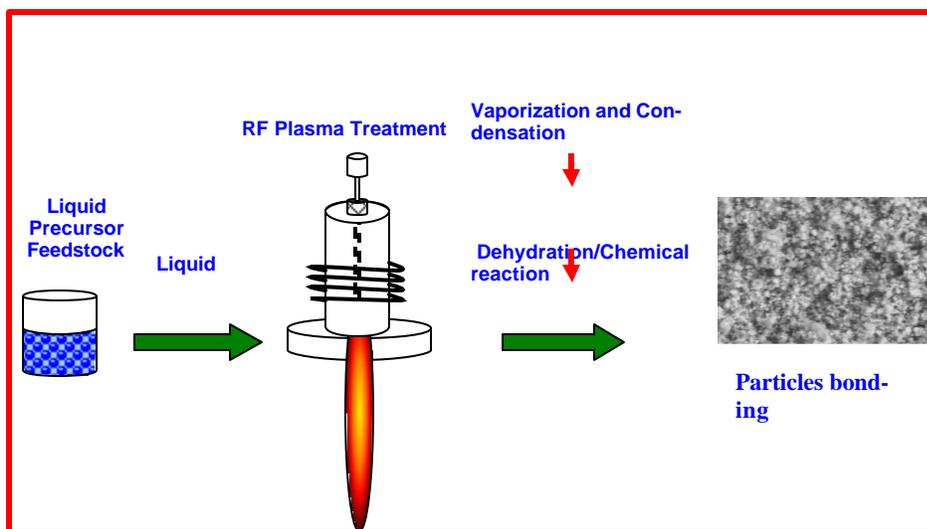


Fig. 2: Schematic of precursor plasma spraying process.

Dr. Andrew Gouldstone Joins CTSR

The *Center for Thermal Spray Research* is proud to announce its newest faculty member, Prof. Andrew Gouldstone.

Andrew received his BS in Materials Science and Engineering from MIT in 1996 and then stayed on at MIT to pursue his PhD in Metallurgy under Professor Subra Suresh. His dissertation was focused on the mechanical behavior of thin metal films and patterned lines on substrates. The first half of his graduate work involved the thermo-mechanical study of such structures, using finite element models and substrate curvature experiments. In the second half, he was introduced to the then-emerging technique of nano-indentation, and used it to explore the incipient plasticity that occurs under small-scale contact. His thesis was capped with an experimental atomic simulation of dislocation nucleation under surface contact, using a variation of the

Bragg-Nye bubbleraft model. Figure 1 captures the essence of the model.

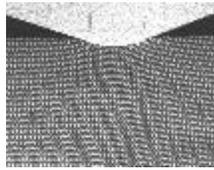


Fig. 1: Demonstration of bubbleraft model.

After graduation in early 2001, Andrew accepted a position as a National Institutes of Health Training Grant Postdoctoral Fellow at the Harvard School of Public Health, studying the lubrication between lung and chest wall during breathing. It was there that Andrew developed an interest in the solid mechanics of lung tissue, specifically the plastic deformation and recovery of lung that occurs in life.

Although respiratory physiology and mechanics remains an interesting topic for him, Andrew was ever drawn back to the

field of materials science, and when the opportunity arose to study the mechanics of thermal spray coatings at CTSR, he quickly accepted the position. Since September 2003, he has had the privilege of working with a number of students in materials, mechanical and electrical engineering, developing robust methods for coating property and residual stress measurement, investigating the nano-scale plasticity of single splats on substrates,



Prof. Andrew Gouldstone

and supporting efforts to determine the fundamental mechanical processes that occur at splat-splat interfaces under macroscopic deformation.

Summer Industrial Training Classes offered By CTSR

Providing a bridge to industry on the latest advances being made within the academic community is one of the key mission's of the CTSR. To accomplish this, the CTSR frequently holds educational courses open to all those interested on a host of topics.

During July 12-13, 2004 full and half day classes ranging from Marketing of TS Technology to Advanced Concepts in TS Materials Processing are scheduled to be offered by Mr. Elliott Sampson, a thermal spray operator and product/marketing manager with over 40 years experience, as well as the faculty and staff of the CTSR.

Class listings include:

Global Opportunities for Thermal Spray Applicators: Thermal spray technology has been utilized for numerous successful industrial applications, but many times people new to the industry, or even those with years of experience, are not aware of these opportunities. This class will provide a strategy to develop and exploit these global opportunities.

Introduction to Thermal Spray Coat-

ings: Those new to the industry in technical as well as non-technical positions can greatly benefit from this course which provides a broad overview of thermal spray technology.

Advanced Concepts in TS Materials Processing: The use of traditional spray parameters for predicting coating properties and performance are clearly not sufficient when process control and reliability are critical issues. The latest techniques for determining in-flight particle characteristics through process mapping and in-situ coating property determination will be explored during this class. Participants will gain a much greater appreciation for the particle science necessary for optimized deposition and coating performance.

Marketing of Thermal Spray Technology: This class is geared towards sales and marketing personnel who can benefit from an improved appreciation of the needs and decision drivers of their customers. Too many times the wrong process or material is specified leaving the customer dissatisfied with the thermal spray process in its entirety. Class per-

ticipants will gain a much better understanding of proper coating selection procedures and economics of the various processing allowing them to make intelligent coating recommendations.

The CTSR is a fully equipped spray and testing laboratory and hands-on training is offered as part of the Introduction to Thermal Spray Coatings and Advanced Concepts classes, making them unique learning experiences.

For additional information, please log onto our website at: www.stonybrook.edu/ctsr



Indentation: A Simple Tool With Powerful Capabilities

Instrumented indentation is well-suited as a technique for probing mechanical behaviour of coatings; it requires minimal specimen preparation, can be performed multiple times on a single specimen, and can measure behavior over various length scales, by recourse to appropriate indenter tip selection. However, the complex nature of the deformation fields under the tip, coupled with complex (e.g. anisotropy, nonlinearity) coating properties necessitates robust means of interpretation in order to extract stress-strain behavior.

At the CTSR, we have developed a simple method, based on empirical work by Tabor, for converting coating indentation force-depth data to stress-strain curves. Using this method, results of indentation can thus be used to quantify mechanical behavior of coatings deposited by different processes (e.g. Fig. 1), and subjected to post-processing treatments, providing a powerful supplement

to microstructural characterization. In addition, we have recently developed a surface marking technique, based on a modified Au sputter system that allows construction of full elastoplastic stress-strain curves using only a Brinell hardness tester (Fig. 2). Such a method allows rapid and objective assessment of coating quality for spray end users. Currently, this method requires polishing of coating surfaces; efforts are underway to extend applicability to as-sprayed materials.

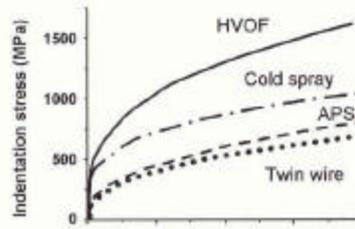


Fig. 1. Stress-strain curves for Ni-5%Al

deposited by four different methods. Elastic portion of the curve is generated via analysis of indentation unloading behaviour and plastic portion is constructed from hardness test data.

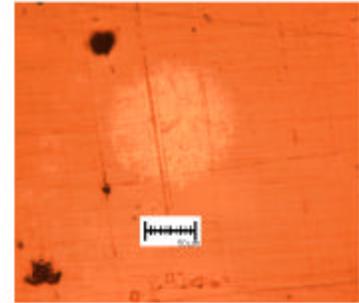


Fig. 2. Deposition of a porous Au film (40 nm thickness) allows determination of the contact diameter resulting from elastic indentation of the coating; all mechanical data can thus be generated from simple hardness testing.

Approaches to Address Process Reliability and Sensitivity

Increasing knowledge in the area of process-microstructure-property relationships allows more precisely specifying desired coating properties and understanding their performance. As a consequence new requirements for process control arise in terms of reproducibility, reliability and predictability to approach the goal of producing coatings according to predefined functionalities. A major step in that direction has been made possible in recent years through a variety of particle diagnostics sensors. At Stony Brook we have taken a successive step to integrate several of these sensors along with in-situ coating property measurements through a multi-dimensional

sensor setup (Fig. 1), which combines simple direct feed back capabilities for online injection optimization with more sophisticated tools for detailed monitoring of in flight particle states as well as in situ coating properties.

A wide range of materials have been investigated through this integrated approach. For example in conjunction with the Stony Brook Industrial Consortium, a detailed study of three different morphologies with nominally the same size distribution process capability for plasma spraying ZrO₂ ceramics was explored (Fig. 2). During 3.5h of gun (7MB) operation, the re-

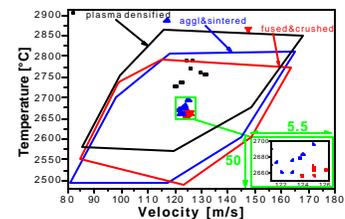


Fig 2: Reproducibility compared to maximum process window.

producibility of a continuously measured central condition (see insert) was evaluated. This study demonstrated overall process window was better $DT=7.3\%$, $DV=3\%$ which was a pleasant surprise. For the more than 21 different spray conditions included in each of the experiments temperature (T) and velocity (V) could be predicted excellently (Chi Square > 90%) from primary parameters. Work is also in progress to examine the relationship between particle state and coating properties. The improvements in process science can be used to develop process maps which will guide parameter selection as well as addressing reproducibility. This is a major step towards achieving prime reliant coatings.

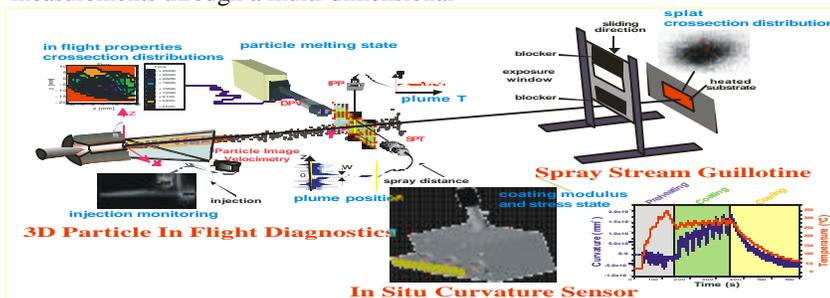


Fig. 1: Sensor set-up to explore process capabilities.

Materials & Processes for Energy Systems Workshop Set for July 14-16, 2004

In honor of Herb Herman, on the occasion of his 70th birthday, a special symposium focusing on Materials and Processes for Energy Systems has been planned for July 14-16th, 2004.

Today's energy systems, and even more importantly those being designed for future applications, are greatly dependent upon high performance surfaces and coatings. Performance thrusts and design requirements for these advanced systems will be discussed by leading applicators and researchers throughout the Symposium. Thermal spray technology has produced successful protective

coatings for decades. The processing of these coatings, however, was generally achieved through Edisonian methods and serendipity. Newer complex materials systems and operational demands have required more sophisticated process control and coating characterization. These and related issues will be the goal of the Symposium.

Scheduled speakers include:

- ?? Subhash Singhal: Solid Oxide Fuel Cells
- ?? Curt Johnson and YC Lau: Thermal and Environmental

Barrier Coatings

- ?? Brij Seth: Design Requirements for High Temperature Oxides
- ?? Fred Pettit: Oxidation in High Temperature Energy Systems (University of Pittsburgh)
- ?? Rich Neiser: Process Science and Control

For additional information or workshop brochure for registration, please log onto www.stonybrook.edu/ctsr/workshops.

Comprehensive Evaluation of TBC Structure Leads to Better Understanding of Structure-Property Correlations

The use of thermal barrier coatings (TBC) to protect high temperature gas turbine systems has clearly become the main stay of the thermal spray industry. Plasma-sprayed yttria-stabilized zirconia (YSZ) TBCs, which yield desirable thermal insulative properties have been the bill-of-materials for this stringent application. Research carried out at Stony Brook jointly with the National Institute of Standards and Technology has focused on determining details (e.g., anisotropic void microstructures) of the imperfection content so that it may be quantitatively separated and thus controlled through the use appropriate process tools. Manipulation of the microstructure offers an opportunity to tailor coating properties to meet application requirement. This requires a clear understanding of correlations between processing, microstructure development and related TBC properties (e.g. thermal conductivity and modulus).

Through the use of advanced scientific tools, the distributions of voids have been delineated and related to specific process parameters. Microstructure and processing variations have been correlated with coating properties. This approach, combined with tracing the evolution of microstructural and property changes for industrially relevant service-encountered heating, is now available to help establish rigorous processing-microstructure-property relationships in future TBC designs.

Recent efforts conducted in concert with GE have focused, on the examination of the critical microstructural features that led to Dense Vertically Cracked (DVC) TBCs, exhibiting quasi-columnar microstructures approximating EB-PVD coatings. Fig. 1 shows the top surface and cross sectional modulus for four such TBCs. Results show a monotonic increase in top surface modulus, however, under cross-sectional indentation, the discrete change in dominant microstructural (porosity) feature is reflected by a transition in measured elastic modulus. The vertical cracks are induced by design; they introduce in-plane compliance that improves resistance to thermal cycling fatigue.

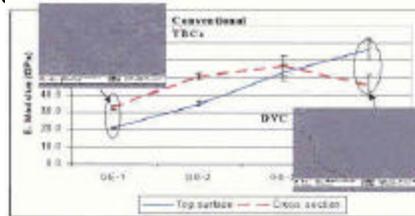


Fig. 1: E. Modulus anisotropy in conventional and advanced TBCs.

The quantitative separation of the microstructure into its components for the two distinct coatings, obtained from small angle neutron scattering, is shown in Fig. 2. It is seen that the component porosity of interlamellar pores decreases, thus enabling a correlation with the thermal conductivity and elastic modulus of the

coatings. Furthermore, the component porosity of intrasplat cracks increase, thereby explaining the decrease in cross sectional modulus for the DVC coating. Thus, the combination of the two measurements in this case provides important information about the multi-functional behaviour of the coating.

Another study involved investigation of the behavior of low, medium and high porous coatings upon isothermal aging. The total void surface area, derived from small-angle neutron scattering measurements, decreases at higher temperatures, and drops sharply above 1100°C, suggesting enhanced sintering of the intrasplat cracks and the inter-lamellar pores. However, a sharp increase in surface area is observed for the 1400°C anneal, this increase can be attributed to a destabilizing of the t' (tetragonal zirconia) phase to the monoclinic phase resulting in a 4% volume change. These new approaches provide new insight into three dimensional microstructural effects and also enable stronger correlation between design relevant properties and the contributing microstructural features.

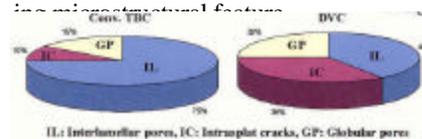


Fig. 2: Quantitative separation of microstructural features using SANS.

CENTER FOR THERMAL SPRAY
RESEARCH

State University of New York at Stony Brook
Department of Materials Science & Engineering
Room 220 Old Engineering
Stony Brook, New York 11794-2275

Phone: 631-632-4567
Fax: 631-632-7878
Email: lysa.russo@stonybrook.edu

Going Beyond the Surface

For More Information:
www.stonybrook.edu/ctsr

Alumni Focus: Dr. S. Rangaswamy, Wall Colmonoy Corporation



own.

Ranga was born and raised in southern India and began his career in engineering at the Hindustan Aeronautics Ltd (HAL) as a supervisor of the tool/die/machine shops. In 1978, Ranga moved to the United States after being accepted at SUNY Stony Brook master's program in engineering. Once Ranga completed his master's degree, he accepted an engineering research and development position with Metco to concentrate on the development of thermal spray materials. During his time with Metco, Ranga published numerous

The Center for Thermal Spray Research is proud to call one of our industry's most prominent industrial scientists, Dr. S. Rangaswamy one of its

technical papers and was granted six US patents for surface coating materials including ceramics, thermal barrier coatings and wear-resistant carbides.

In 1983 Ranga returned to SUNY Stony Brook to pursue his doctorate degree while still working part time at Metco. In his Ph.D thesis, "Metallurgical Characterization of Plasma Sprayed WC-Co Coatings", Ranga explored the metallurgical reactions in tungsten-carbide-cobalt materials during plasma spray operations. His research was one of the first semi-quantitative studies of these metallurgical reactions and helped enable designers of thermal spray powders and systems to design them for optimal performance.

Ranga left Metco in 1988 and moved to Michigan to join Sulzer (now Sulzer Metco). While at Sulzer, Ranga was granted eight more patents and authored numerous technical papers and presentations on thermal spray technology.

One of Ranga's most significant patents was for a new composite pow-

der manufacturing process which eliminated the need for binders used to hold the powder particles together. This patent earned Ranga the Sulzer Corporation Innovation Award for Technology.

Ranga joined Wall Colmonoy in February 1996 and as the Director of Technical Services and Corporate Quality is responsible for all of the R&D, Technical Services and Quality Assurance for the entire corporation.

Though Ranga has been able to claim such great successes with his career, he is not all work! He and his wife, Geetha, have been married for nearly 31 years and in November of 2004, they will have the wonderful pleasure of celebrating the marriage of their daughter Sowmya.

Ranga also enjoys drawing, acting and music and performs in traditional Indian plays performed in his native tongue, Tamil. Ranga is also active in organizing local music festival and concerts in south Indian classical music.