PENNSTATE ARL

iMAST

Institute for Manufacturing and Sustainment Technologies

A U.S. Navy Manufacturing Technology Center of Excellence

QUARTERLY 2002 No.2

A Visit by the Chief of Naval Operations

Admiral Vern Clark, 27th Chief of Naval Operations, visited the Applied Research Lab recently as part of a capabilities overview. As Chief of Naval Operation (CNO), Admiral Clark is the senior military officer in the Department of the Navy. Admiral Clark is responsible to the Secretary of the Navy for the command, utilization of resources and operating efficiency of the operating forces of the Navy and Navy shore activities assigned by the Secretary. A member of the Joint Chiefs of Staff, the Admiral Clark is the principal naval adviser to the President and to the Secretary of the Navy on the conduct of war. He also serves as principal adviser and naval executive to the Secretary on the conduct of activities within the Department of the Navy.

iMAST Repair Technology (REPTECH) program manager, Sean Krieger (left), shows Admiral Vern Clark a rotorcraft blade paint stripping sample while ARL Director, Dr. Ray Hettche, looks on. iMAST's Repair Technology effort is a unique Navy ManTech Program established to support Navy and Marine Corps depots and shipyards facilities.

As part of his visit, Admiral Clark received a detailed review of iMAST's repair technology program. In follow-up correspondence to Penn State, the admiral noted: "The briefs from the Applied Research Laboratory were nothing short of impressive and provided me exactly what I needed. It's clear to me that the work being done there is exactly what the Navy needs as we face the challenges of Transformation."

A native of Sioux City, Iowa, Admiral Clark graduated from Evangel College. The admiral also holds an MBA from the University of Arkansas. Following commission via OCS in August of 1968, the admiral served as a surface line officer aboard various destroyers. After selection to flag rank, Admiral Clark commanded the Carl Vinson Battle Group/Cruiser Destroyer Group Three, the Second Fleet, and the United States Atlantic Fleet. In shore assignment, Admiral Clark served as special assistant to the Director of the Systems Analysis Division in the Office of the Chief of Naval Operations. He later completed assignments as administrative assistant to the Deputy Chief of Naval Operations (Surface Warfare), and administrative aide to the Vice Chief of Naval Operations. He served as Head of the Cruiser-Destroyer Combat Systems Requirements Section and Force Anti-Submarine Warfare Officer for the Commander, Naval Surface Force, U.S. Atlantic Fleet. He directed the Joint Staff's Crisis Action Team for Desert Shield and Desert Storm.

Admiral Clark served as Director of Plans at the U.S. Transportation Command holding both Plans and Policy (J5) and Financial Management and Analysis (J8) billets. While commanding the Carl Vinson Battle Group, he deployed to the Arabian Gulf and later served as the Deputy Commander, Joint Task Force Southwest Asia. Admiral Clark has also served as the Deputy and Chief of Staff, United States Atlantic Fleet; the Director of Operations (J3) and subsequently Director, of the Joint Staff.

Focus On Materials Processing

Report Documentation Page

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PENNSTATE
iMAST
Applied Research Laboratory
Institute for Manufacturing and Sustainment Technologies

DIRECTOR
Robert B. Cook
(814) 863-3880 rbc5@psu.edu

MATERIALS PROCESSING and DRIVETRAIN TECHNOLOGIES
Maurice F. Amateau, Ph.D.
(814) 863-4481 mfa1@psu.edu

LASER PROCESSING TECHNOLOGIES
Richard P. Martukanitz, Ph.D.
(814) 863-7282 rxm44@psu.edu

ADVANCED COMPOSITES MATERIALS TECHNOLOGIES
Kevin L. Koudela, Ph.D.
(814) 863-4351 klk121@psu.edu

MANUFACTURING SYSTEMS TECHNOLOGIES
Mark T. Traband, Ph.D.
(814) 865-3608 mtt1@psu.edu

NAVY/MARINE CORPS REPAIR TECHNOLOGIES
Sean L. Krieger
(814) 863-0896 slk22@psu.edu

iMAST ADMINISTRATOR and EDITOR
Gregory J. Johnson
(814) 865-8207 gjj1@psu.edu

STAFF ASSISTANT
Lori L. Mowery
(814) 865-3264 llm1@psu.edu

WORLD WIDE WEB
www.arl.psu.edu/areas/imast/imast.html

NAVY PROGRAM MANAGER
James G. Mattern
(202) 781-0737
matternjg@navsea.navy.mil

IN THIS QUARTERLY

DIRECTOR'S CORNER

Implementation, implementation, and implementation

In the world of ManTech, the adage continues to hold true that implementation is the best measure of program success.

What can you do to maximize your chances of success? First and foremost, you must have clearly defined requirements from the stakeholder. Recognize that the requirements may change over the life of the program, so constant communication within the team is crucial to meeting the objectives of the project. Meet the requirements without "gold-plating," as an unaffordable success is of the same value to the stakeholder as a technical failure.

Second, you must make affordability a major focus. To convince a program manager to spend resources to institute your process, it must be affordable. You should have some visibility into the costs that the program office incurs to implement your process. Often, the costs required to implement a process far exceed the costs for research. How can you mitigate the costs for implementation? You can piggyback on existing testing to certify your process. You can time your change with other planned improvements, thus reducing the costs for revising the logistics. You can team with the contractor on the process, ensuring your development is compatible with existing equipment, or by ensuring the return on investment is attractive enough to make the change.

Third, you must meet the stakeholder's schedule. Windows of opportunity are small and of short duration, and may change. By keeping the communication lines open, you should be aware of changes. With some luck and skill, you could adjust your schedule to meet the needs. Again, for the sake of affordability, meeting testing windows can make the difference between success or failure.

Not every project will be implemented. Improvements in competing processes may reduce the return on investment to below acceptable limits. The project may fail to meet all the necessary requirements. During periodic reviews, it is probable that a project can be re-scoped or even canceled if the prospective for implementation is slim.

This newsletter features our efforts dealing with electron beam-physical vapor deposition of rhenium. While this process is deemed a technical success, the project has been terminated because of changes in the acquisition program.

As we go to press, we are putting our "issues" together for the FY-03 input. If you have any challenges that you'd like to discuss, please give me, Sean Krieger or Greg Johnson a call to discuss. Remember that we need to bring together a Navy-Marine Corps program office, depot or shipyard to close the loop.

As always, we invite your feedback and questions about this newsletter or any issues pertaining to iMAST's mission. We are here to support industry and government. We take our value-added "honest broker" status seriously. We have great talent waiting to support you.

Bob Cook

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Focus on Materials Processing

Net-Shaped Fabrication of Rhenium Components by EB–PVD

by Jogender Singh and Doug Wolfe

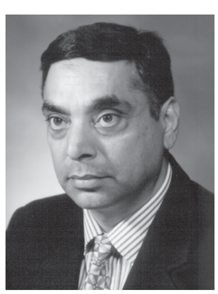
Rhenium, as a pure refractory metal, is a very attractive material for high-temperature structural and energy system applications such as solar-powered rocket engines, heat exchangers, and space and missile propulsion systems. Rhenium has many advantages over other candidate materials including tungsten. It offers excellent erosion resistance for components in high-temperature rocket engines and hot gas-valves. Rhenium has the second highest melting temperature next to tungsten. Unlike tungsten, it has a ductile-to-brittle transition temperature well below room temperature. Among the refractory metals, rhenium has the greatest tensile and creep-rupture strength at elevated temperatures. Rhenium cold work hardens and may only be worked 5–10 percent before requiring high-temperature annealing to full recrystallization. Due to its unique high-temperature physical and mechanical properties, the U.S. Navy has selected rhenium material for the solid divert attitude control system (SDACS) of the SM3 missiles program. Various components including thrusters, pilot valve tubes, gas supply tubes, strainers, rhenium coated graphite balls are needed to be manufactured for the SDACS unit.

It is very difficult to manufacture net-shaped components made of refractory metals, i.e., rhenium. Typically, components are fabricated by either powder metallurgy (P/M) or chemical vapor deposition (CVD). Due to difficulties encountered in the P/M fabrication and shaping of refractory parts, CVD is mainly used in fabricating thin walled, small diameter or complex shaped components and also for coatings on carbon, ceramic and metal components. However, CVD process also has many shortcomings. For instance, rhenium deposition is obtained by passing chlorine gas through a heated chamber (at 500°C) containing rhenium chips resulting in rhenium penta-chloride (ReCl5). The gaseous molecules of ReCl5 decompose at 1200°C with rhenium atoms depositing on the substrate. CVD rhenium deposits contain entrapped gases (chlorine and hydrogen) as impurities resulting in lower physical and mechanical properties. CVD coating often produces columnar microstructure that is undesirable for structural applications. The columnar microstructure is destroyed by periodically taking out the partially coated components followed by mechanical grinding, i.e., partially removing rhenium coated material and continue recoating. This effort is repeated many times to obtain desired thickness and density of the coating. It has been reported that the CVD rhenium substrate produced in this way exhibits multiple layers. When the CVD Re component is heated to elevated temperature, individual layers tend to separate, allowing slippage. This phenomenon has shown to lead to some variability of CVD Re mechanical properties, which is highly undesirable for the design and incorporation of flight engines.[1, 2] The second shortcoming of the CVD process is that it requires long lead-times in fabrication of components, and thus it is not a cost effective manufacturing process.

Since CVD is not a line-of-sight process, interior and exterior of the complex part can be coated simultaneously. However, it is difficult to apply uniform coating ‘thickness’ on spherical components, i.e., graphite balls due to limited flexibility in maneuvering parts in the reaction chamber, gas flow dynamics and entire surface area to be coated simultaneously (i.e, 360°). Depending the dimension of the components, generally one component (such as thruster) is manufactured at a time in the CVD reactor chamber.

Powder metallurgy techniques have been explored in the fabrication of rhenium components.[3] These techniques also have their limitations with respect to cost, speed, achievable geometry, required tooling and high temperature isostatic pressure (HIP) treatment for compaction.[4] Various steps are involved in the manufacturing of components including cold isostatic pressing, pre-sintering at 1200°C, hot sintering at 2500°C. Density of greater than 99 percent is achievable only after extensive amounts of accumulated cold and hot working. Hot working of rhenium must be carried out in a hydrogen environment. In air, the rhenium metal readily oxidizes to the heptoxide (melting point 297°C), so hot working in the air is not possible. This extensive value-added

PROFILE



A respected leader in his field, with research experience in academia, industry and national labs, Jogender Singh is a senior scientist with ARL Penn State. Dr. Singh’s expertise includes coatings and surface modification of materials by various techniques including laser and the EB-PVD process. He is actively working in the synthesis of nano-particles and characterization by various techniques including SEM, X-ray, and TEM.

Prior to joining Penn State, Dr. Singh worked for NASA and GE in development of new materials and coatings for aerospace applications. His interests continue to be surface engineering and technology enhancement for various applications. Dr. Singh is a Fellow of ASM, FIM, and FAAAS. He can be reached at (814) 863-9898, or by e-mail at: <jxs46@psu.edu>.

processing contributes to high cost and fairly limited range of commercial shape components.[4] Fabrication of components by powder-plasma spray process has also been explored and exhibited poor mechanical properties due to presence of large volume fraction of porosity.

An effort was undertaken to address shortcomings of CVD and P/M techniques and identify an alternative manufacturing method in fabricating net shaped components with density greater than 95 percent in a cost-effective manner. Electron beam-physical vapor deposition (EB–PVD) method has been explored to meet challenges and goals of the SM3 program. EB–PVD is one of the oldest technologies used in applying ceramic and metallic coatings for a wide range of application from microelectronics to turbine industry. EB–PVD is a matured technology and it is a robust process. EB–PVD is currently being used by many industries including IBM, GE, and Pratt & Whitney in their product field.

Physics of EB-PVD

Physics of coating deposition mechanism is very simple in the EB–PVD process, i.e., material is evaporated by high-energy focused electron beam in a vacuum chamber, and the evaporated molecules condensate on the substrate (Fig. 1a).

Formation of coating occurs involving two steps, i.e., nucleation followed by subsequent growth of the condensate molecules as shown in Fig. 1b. Physical vapor deposition is primarily a line-of-sight process; therefore, uniform coating of complex parts is accomplished by continuous rotation in the vapor cloud during evaporation process. Deposition rate and coating thickness depends on the material evaporation rate, deposition time, chamber pressure, and operating power of the electron-beam guns.

EB–PVD Equipment

Penn State has a world-class coating research facility with two EB–PVD units and sputtering. The first unit is the ion beam-assisted EB–PVD system, which has one EB gun (8 kW), four 25 cc hearths, and a cold cathode ionization source; the chamber size is 66 cm × 60 cm × 100 cm. In addition, this unit has a real-time coating thickness measurement capability during the deposition process as well as gas analysis and substrate bias features. These features are advantageous for process optimization, modeling, and controlling the microstructure of the coating. This unit is used for the initial development of coatings.

The second unit is an industrial prototype EB–PVD unit that has six electron beam guns (Fig. 2). Four EB-guns are used to evaporate coating materials and two EB-guns are used to preheat the substrate to facilitate coating adhesion. Each gun has an average 45 kW power. The chamber accommodates up to three ingots 7 cm in diameter and 50 cm long. Overall, the chamber is approximately 90 cm long, 90 cm wide, and 90 cm high. The maximum diameter of the substrate (with vertical rotation) that can be accommodated is about 40 cm. Parts can be manipulated in three dimensions on a computer-controlled rack system at speeds of 5.5–110 rpm and with a maximum load of about 100 Kg.

Advantages of the EB-PVD Process

The EB–PVD process offers extensive flexibility in controlling composition and microstructure of the coatings. Composition variation is accomplished by independent evaporation of materials from multiple ingots. This process has the flexibility to integrate multilayered ceramic-metallic coatings on components. The deposition rates of the coating range up to 1–150 μm/minute while the total coating thickness can range from 1 micron to greater than 2 cm in thickness. Parts to be coated are heated directly by oscillating electron beam guns or indirectly using graphite plates. Coatings produced by the EB–PVD process usually have a good surface finish and a uniform microstructure. Manipulating the process parameters and ingot compositions can easily alter the microstructure and composition of the coating.

Figure 2. Electron Beam–Physical Vapor Deposition unit.

Advantages of Ion Beam-Assisted Deposition

Typical energy of the vapor cloud produced by thermal or electron beam evaporation is about 0.1–1 eV. This energy is not sufficient to have good atomic mobility during condensation on the substrate. Bombardment of the ionized gas on the substrate surface enhances the atom mobility of the condensate. Thus, attachment of an ion beam-assisted source to the EB–PVD offers additional benefits such as forming denser coatings with improved microstructure and good metallurgical bonding with the substrate. In addition, textured coatings are obtainable which is desirable in many applications. The state of the internal stresses can be changed from tensile to compressive by ion bombardment with energies ranging from 10–100 eV. Thus, the ability to control stress levels in multilayered coatings is an

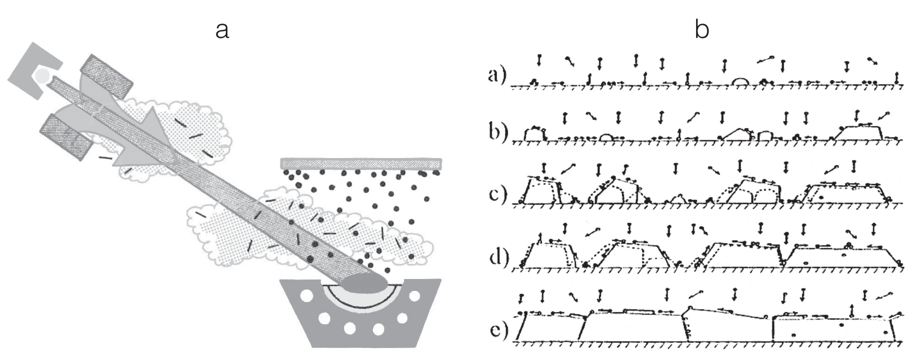


Figure 1. Principal of EB-PVD technology and coating formation mechanism.

additional advantage of the ion beam-assisted process. A high-energy ion beam (as a source of energy) is quite often used to clean the surface of the component inside the vacuum chamber prior to coating. This enhances the metallurgical bonding strength between the coating and the substrate.

Rhenium Plate Fabrication

Efforts were undertaken to demonstrate that rhenium plates produced by EB–PVD would have equivalent or better physical and mechanical properties to those made by conventional CVD processes. The rhenium ingot that was used as source material was supplied by Rhenium Alloys, Inc. (density 98 percent, purity 99.99 percent). A focused high-energy electron beam was used to evaporate the rhenium ingot in the coating chamber and deposit it on graphite plates. During the deposition process, the graphite plates were indirectly heated up to 1000°C. Coated plates exhibited higher hardness (283 VHN) in comparison with CVD rhenium plate with hardness 245 VHN. Using the Hall-Patch equation along with the grain size and hardness, it is predicted that the rhenium plate (Re-plate) will exhibit 30 percent improvement in mechanical properties as compared with CVD Re-plate. Rhenium plates were found to be free from impurities such as copper or other materials from vacuum chamber. A novel concept was developed to avoid any continuous columnar grain formation during deposition process (Fig. 3).

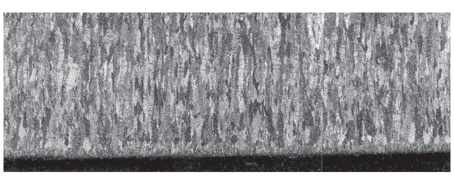


Figure 3. Optical micrograph showing layered deposit of 40 mil thick rhenium plate by EB–PVD with tensile strength 72 KSI and hardness 283 VHN. Typical tensile strength of CVD Re is 50 KSI and hardness 245 VHN.

In addition, rhenium plate exhibited textured grain growth with micron and sub-micron sized microstructure. Coefficient of thermal expansion (CTE) of the EB–PVD Re-plate was comparable with the CVD Re-plate. The EB–PVD Re-plate was produced by conventional method, i.e., without using ionized gas bombardment. It is anticipated that the microstructure of the Re-plate produced by ion-beam assisted EB–PVD would exhibit much finer grain microstructure with superior mechanical properties.

Rhenium-Coated Graphite Ball Fabrication

Applying uniform rhenium coating on graphite balls (or cores) is another success story. About 18 graphite cores were simultaneously charged into a cylindrical cage as shown in Figure 4a. The cage was fabricated using molybdenum wires and plates. The cylindrical cage was rotated at 7–10 rpm above the melt pool in the rhenium vapor. During deposition process, cores were heated to 1000°C and simultaneously bombarded with ionized argon gas to obtain dense microstructure. After applying rhenium to the full coating thickness, eighteen coated cores were simultaneously polished in the laboratory vibromet-polishing unit to the surface finish <Ra8 (Figure 4b). All coated cores exhibited uniform coatings with 100% concentricity, which was measured by coordinate measuring machine (CMC). It is important to mention here that there are more than 250 micro and sub-micron grains through the coating thickness (Figure 5) with much finer grained structure than the current CVD process. It has been projected that the cost of manufacturing rhenium-coated cores by EB-PVD would be less than 50 percent of the current CVD process.

In the CVD process, 2–4 graphite cores are coated simultaneously with periodic changing the angle of rotation with respect to the flow direction of the rhenium molecules. It has been well documented that the rejection rate is very high (greater than 75%) due to concentricity and non-uniform coating thickness. In addition, there are only 2–5 grains through the 30 mil coating thickness.

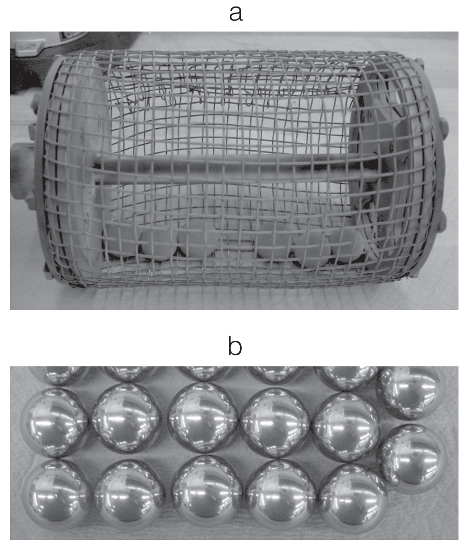


Figure 4. (a) Cage used for applying Re coatings on 18 graphite cores and (b) polished cores with surface finish <Ra8.

Fabrication of Rhenium Tubes

A similar effort was undertaken in the manufacturing of pilot valve rhenium tubes with a wall thickness 10 mil and lengths of 8–10 inches. Such tubes are manufactured using molybdenum (Mo) tubes as a mandrel on which rhenium is deposited. The Mo mandrel is removed by chemical dissolution leaving behind the skin of the coating, i.e., rhenium in the tubular form. Currently, Re tubes are manufactured by CVD. The main drawback of this process is that the Re tube contained 2–4 grains through the wall thickness does not provide adequate number of grains for welding and bending applications (Fig. 6). In addition, there are not enough grains through the wall thickness to accommodate pressure under high-temperature burst tests. Again, CVD process is limited in manufacturing of tubes (2–3 at a time).

The shortcoming of CVD process was addressed by exploiting EB–PVD technology. Similarly, Mo mandrel concept was used in manufacturing of Re tubes. Tooling concept developed in the manufacturing of Re tube by EB–PVD is displayed in Fig. 7. Eleven Mo mandrels were mounted simultaneously for applying Re coatings. Mo mandrels were periodically rotated to get uniform coating thickness across the diameter. Mo mandrels were heated indirectly to a

b

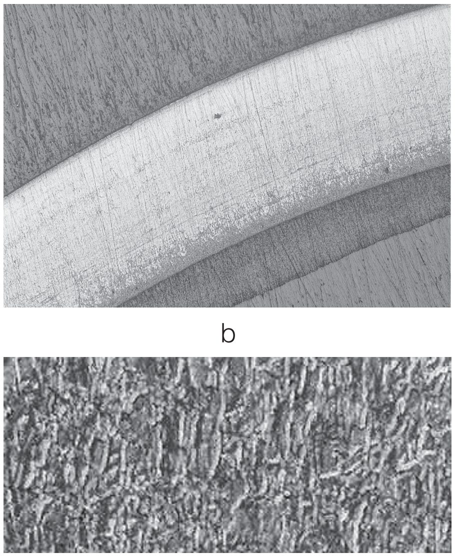


Figure 5. Cross-section of the rhenium-coated core exhibiting dense coatings with nano and submicron-sized grained microstructure (a) low magnification (b) high magnification.

1000°C during Re deposition.

The cross section of the rhenium-coated Mo mandrel is shown in Fig. 8. Rhenium coating was very uniform around the Mo tube up to 8 mils deposition. The last segment of the Re deposition exhibited non-symmetrical coating due to tooling rotation failure. However, optical microstructure exhibited more than 40–50 grains through the wall thickness that is 10 times more than the current CVD process.

Fabrication of Net-Shaped Thruster

Net-shaped thruster fabrication by EB–PVD was demonstrated using titanium as an evaporant material instead of rhenium. Due to high flexibility in the EB–PVD process, two thrusters (mirror image) were made at the same time during deposition process (Fig. 9). Results were very promising with uniform coating thickness along with the graphite mandrel. Efforts are underway to duplicate similar effort using Mo-mandrel and Re as an evaporant material. In contrast, rhenium thruster is currently made by CVD process and only one thruster is made at a time due to limited flexibility in the chamber.

Summary

EB–PVD has demonstrated to be proven

a

b

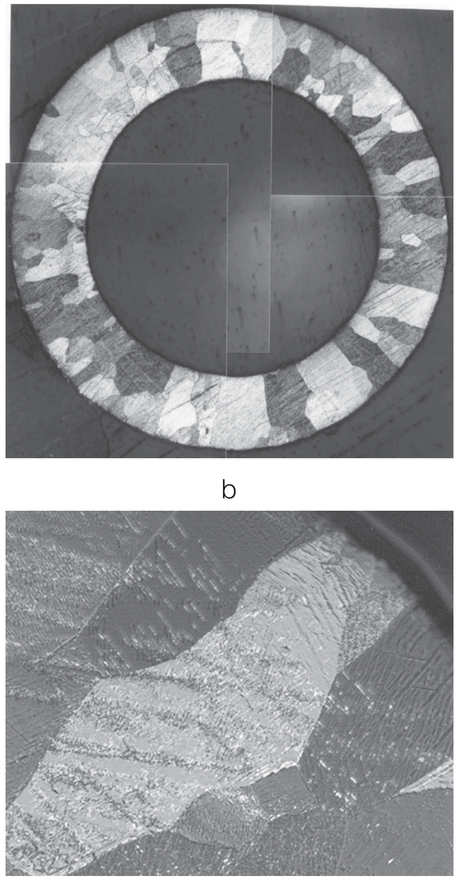


Figure 6. Cross section of rhenium tube produced by CVD exhibiting 1–3 grains through 10 mil wall thickness (a) low magnification and (b) high magnification.

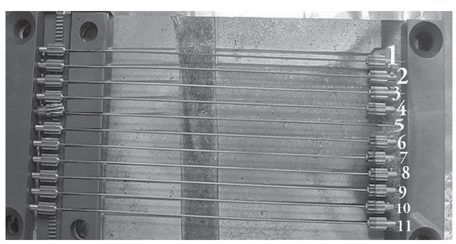


Figure 7. Tooling used in the fabrication of eleven rhenium tubes simultaneously.

technology in the fabrication of net shaped components in a cost effective manner with superior microstructure and mechanical properties. It is predicted that the cost of rhenium components manufactured by EB–PVD process would be 50 percent less as compared to current CVD and powder-HIPing process. This is a robust process with a high degree of flexibility in controlling composition and microstructure of the components. Unlike CVD process, no intermediate machining is required in destroying columnar microstructure, rather much

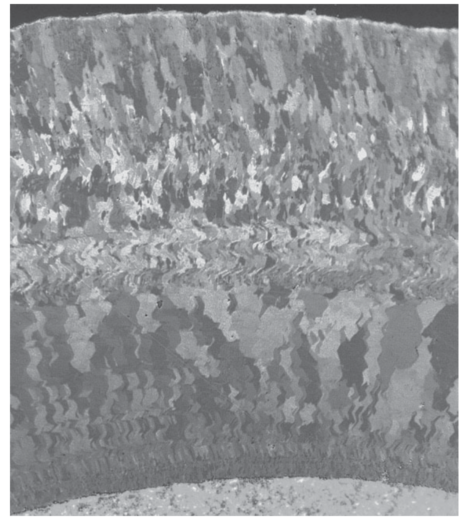


Figure 8. Cross section of rhenium tube produced by EB–PVD exhibiting greater than 40-50 grains through 10-mil wall thickness.

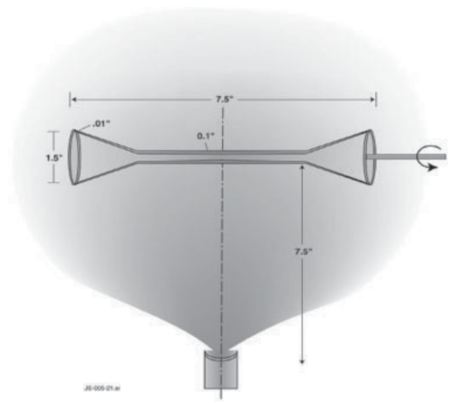


Figure 9. Simulated titanium coated graphite mandrel thruster. Advantages: High production rate with tailored microstructure.

finer grained microstructure is achievable in the EB-PVD. Unlike powder metallurgy processes followed by HIPing, no surface machining is required to get desired surface finish. This is a one step process.

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4. J. O. Milewski, D. J. Thoma, J. C. Fonseca and G. K. Lewis "Development of near net shape processing for rhenium using directed light fabrication" *Materials and Manufacturing Processes*, Volume 13 (1998), pages 719-730.

(left to right) ARL's Tom Donnellan and Bob Cook pause with exhibit booth visitor Robert Williams, program manager for Boeing Phantom Works Advanced Army and Rotorcraft Programs.

Tom Donnellan (left) Bob Cook (rear left) and Russ Burkhardt (rear right) look on as Dr. Ray Hettche discusses technical aspects of ATT prototype with Congressman John Murtha.

iMAST's Greg Johnson discusses aerospace capabilities with Dr. Chris Brackbill, an aerospace engineer with U.S. Army Aviation and Missile Command, at the Penn State AHS 58 booth.

Tech Trends 2002

iMAST, as part of an ARL Penn State contingent, recently participated in Tech Trends 2002 at the convention center Baltimore. The Tech Trends conference series is a forum for interested parties within the Mid-Atlantic states (PA, NJ, DE, and MD) who want to learn about federal research and development programs in such diverse areas as electronics and computers, medical and pharmaceutical research, aeronautics, space, national defense, homeland security, energy and environmental technologies. The conference is intended to provide opportunities for researchers and executives to engage in dialog with representatives from government R&D agencies as well as major R&D corporations.

Johnstown Showcase for Commerce

The annual Johnstown Showcase for Commerce was once again the site of an iMAST exhibit booth. Held in Cambria County, Pennsylvania, the showcase provides an opportunity for business, government and research organizations alike to showcase advanced technology efforts being developed throughout the local region. Industry support by DRS, United Defense, Raytheon, General Dynamics and The Boeing Company attest to the quality level of participation. Forums like the Johnstown Showcase for Commerce aid technological innovation efforts by bringing together the necessary ingredients to transfer technology into both civilian and DoD manufacturing sectors. opportunities like this showcase provide smaller organizations an excellent chance to interface with key players in the research and developmental world, as well as manufacturers and DoD customers. In many cases, out-of-the-box thinking emerges as dialog develops between researchers, manufacturers and customers. The annual Johnstown Showcase for Commerce is scheduled again for next June. Check our calendar of events in future newsletters for more information.

Annual American Helicopter Society Forum

Hovering just over the border of Canada, the American Helicopter Society held its second out-of-country forum. The grand city of Montreal hosted the 58th annual forum. This year's theme "Vertical Flight Technology: Building a Global Consensus" drew a good industry crowd as well as military representatives from NAVAIR and the U.S. Army. Dr. Nagesh Sonti, of ARL Penn State's Drivetrain Technology Center, provided an overview on the on-going ausform gear finishing effort. Next year's forum will be held in Phoenix, Arizona (May 6-8). For more information about AHS call (703) 684-6777 or write Mr. Rhett Flater, Executive Director AHS, 217 N. Washington Street, Alexandria, Virginia 22314 or e-mail him at: <ahs703@aol.com>.

Proceedings Article

iMAST's Bob Cook and Greg Johnson recently teamed up with Brigadier General James Feigley, USMC (Commanding General, Marine Corps Systems Command) to discuss technology and the U.S. Navy ManTech Program in the June 2002 issue of *Proceedings*, published by the U.S. Naval Institute. A copy of the article is located on-line in the publications section of the iMAST web site. You may visit this site at: <www.arl.psu.edu/areas/imast/imast.html>

CALENDAR OF EVENTS

31 July–2 Aug.	ARMTech Showcase		Kittanning, PA
13–14 Aug.	3rd Annual ONR Naval-Industry R&D Conference	★★★★★ *visit the iMAST booth*	Washington, DC
19–23 Aug.	Penn State Rotary Wing Technology Short Course		University Park, PA
10–12 Sept.	ASNE Symposium		Bremerton, WA
17–19 Sept.	Marine Corps League Expo	★★★★★ *visit the iMAST booth*	Quantico, VA
Sept. TBA	NDIA Tracked Vehicle Conference		Ft. Knox, KY
21–24 Oct.	NDIA Expeditionary Warfare Conference		Panama City, FL
29–30 Oct.	DoD Maintenance Conference 2002		Reno, NV
13–14 Nov.	Materials and Manufacturing Advisory Board meeting		State College, PA
2–5 Dec.	Defense Manufacturing Conference 2002	★★★★★ *visit the iMAST booth*	Dallas, TX
2003			
TBD Mar.	Navy League Expo		Washington, DC
TBD Apr.	Tech Trends 2003	★★★★★ *visit the iMAST booth*	TBA
6–8 May	American Helicopter Society Forum	★★★★★ *visit the iMAST booth*	Phoenix, AZ
16–17 Jan.	ShipTech 2003		Biloxi, MS

Quotable

"When we go to war, we never want to have a fair fight."
—James Roche, Secretary of the Air Force

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