# Newsletter





Institute for Manufacturing and Sustainment Technologies

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#### **Calendar of Events**

#### **DIRECTOR'S CORNER**

After a very long winter I am happy to report that the iMAST team is recovering from vitamin D deficiency. Yes, the sun came out here—finally!

In case you are wondering, iMAST has survived the Continuing Resolution and Sequestration... at least so far. We are hopeful that the great work



Timothy D. Bair

being performed by our project teams at ARL, and the great track record we have successfully implementing our technology, will continue to make this relationship durable and lasting.

As of this publication we're rabidly working to identify new projects to carry us well into FY-15 and beyond. As in years past, if you have a good idea for a project that makes our ships cheaper to acquire or any of the Department of the Navy's weapons platforms easier and cheaper to operate over their life cycle, give us a call.

**Continued on Page 2** 

# Surplus Engines Provide Multiple Building Points for iMAST Activities

Recently, iMAST was able to obtain two surplus T53 turbine engines from the U.S. Army via the Commander, U.S. Army Aviation and Missile Command at Redstone Arsenal in Alabama. These engines will be used in association with various Navy ManTech projects that address coatings, complex systems monitoring, and materials science efforts currently underway at Penn State's Applied Research Laboratory (ARL).

Dr. Doug Wolfe, senior research associate in ARL's advanced coatings department notes: "The T53 engines donated by the U.S. Army will be used to assist in training our

next generation of scientists, engineers, undergraduate and graduate students in the field of material science and engineering. These efforts include extending the life of engine components, to further improving



engine efficiency and performance. We plan to use select engine components/blades and apply advanced erosion and corrosion resistant coating materials and material systems and assess the performance. Other engine components will be used to assist in designing advanced coating material manufacturing processes." Dr. Tim Eden, ARL's Materials Processing Division head, adds: "The T53 engines additionally allow faculty and staff opportunities to further develop additive manufacturing processes for the production of specific parts for aerospace application. These processes include Laser, Cold Spray and E-beam processes. Wear resistant coatings, including a metal/solid lubricant composite that can reduce fretting in jet engines are being deposited using the Cold Spray process. The opportunity to have a turbine jet engine available is a significant plus relative to our process development efforts."

> In addition to the value the acquisition of these engines provides, significant disposal costs were avoided in the process of the procurement.

> In addition to the research-oriented

efforts these engines will support, we are hopeful the engines will spark interest within student interns relative to them considering career paths in the various Army and Navy systems command sectors. As the civil service workforce sector grows gray, it is imperative that recruitment efforts on behalf of the various service branches attract the best and the brightest.



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# **DIRECTOR'S CORNER**

That said, in all of our projects, implementation success is the goal, along with a positive return on investment for the ManTech effort. If it doesn't have a good chance of ending up on a ship, aircraft or ground combat vehicle-we don't do it. Occasionally we cancel projects (in their mid-term) that do not look promisingtransitionally. Reducing risk is part of the DoD ManTech directive. That said, when it is apparent that the risk is not going to pay off, we programmatically back away to pursue other avenues of approach-which will successfully address the various challenges. This is part of our duty to the taxpayer. On the other hand, successful projects that end, but need more push to transition through the technology "Valley of Death," will continue to get attention and emphasis from us until it is a total success. Our track record reflects this dogged determination in our work-and our reputation.

Our feature article in this newsletter issue is one example. About 5 years ago we were asked to evaluate, as a trusted agent, the state of the technology in rapid testing and identification of hazardous materials in coatings. The project was a success but the findings determined that the "state" was insufficient for public shipyard applications at the time. More recently, NAVSEA renewed their request to again evaluate the state of hazardous, heavy metal detection across the Navy's spectrum of platforms in the repair and maintenance process. This time iMAST investigators determined that indeed, the technical capability had matured and could be applied to shipyard and depot overhaul operations. Transitioning the process to the shipyard is complete but implementing it into the various fleet efforts is still a work in progress. We are supporting Norfolk Naval Shipyard as they continue to complete this particular effort. This is yet another example of the great work being done by the iMAST-NAVSEA team.

As I close, I invite you to review our calendar of events in order to identify opportunities where you can possibly interact with us in geographic areas convenient to you. And it goes without saying, if you think you will be in our neighborhood, please don't hesitate to coordinate a visit with us. Please contact us if you have any questions. We continue to solicit your ideas, concerns, challenges and input.



## PROFILE



Janice Keay is an Associate Research Engineer at Penn State's Applied Research Laboratory. Since 1993, Ms. Keay has been employed in the Materials and Manufacturing Office's Manufacturing Systems Division (Environmental Technology Group). Ms. Keay's body of work has focused on environmental research studies, as well as air and water analysis. Ms. Keay's various projects have included determination of the capture efficiency of a paint overspray

shroud, studies on the photolytic degradation of volatile organic compounds, and development and testing of handheld HAZMAT analyzers for paint analysis.

Ms. Keay earned her B.S. in chemistry from Purdue University and an M.S. in chemistry from Fairleigh-Dickinson University. Ms. Keay has over thirty-five years of experience in analytical chemistry, particularly chromatography and spectroscopy.

For more information on Ms. Keay's featured article, please contact Dan Merdes, Ph.D. at (814) 863-4145, or by e-mail at <dwm@arl.psu.edu>.



## Focus on Manufacturing Systems

# Heavy Metal

#### by Janice Keay

A portable COTS (Commercially-available Off-The-Shelf) X-ray Fluorescence (XRF) analyzer has been evaluated, validated, and demonstrated at Norfolk Naval Shipyard and found to be capable of meeting shipyard requirements for the ship-side analysis of heavy metals in paint. Portable XRF analysis of lead, chromium, and cadmium in paint meets the shipyard specified and required needs for safety, for analytical accuracy and sensitivity, for improved on-demand reporting of results and for practical adaptation to use in the shipyard environment. The XRF sample preparation and testing takes about 10 minutes per sample and 25 samples can be tested, with results provided to the paint shop, in less than 4.5 hours.

#### **HISTORY**

In situ analysis of heavy metals in paint has been a shipyard issue discussed at RepTech Working Group meetings for more than 14 years; two earlier RepTech projects, in 1998 (Sames) and in 2005 (Keay), investigated Laser Induced Breakdown Spectroscopy (LIBS) for its potential application to this problem, but LIBS was not sensitive enough to make the technology suitable. Technology reviews in the late 1990s and early 2000s into the possible application of point-and-shoot X-ray Fluorescence (XRF) analysis for the in situ analysis of heavy metals in paint on ships found that the analyzers would not meet shipyard needs for several reasons, including: the use of a radioactive source to generate the X-rays, detection levels for chromium that were not sensitive enough and that were marginal for lead, and test results (reported in units of surface area, µg/cm<sup>2</sup>, rather than ppm) that could not be compared to current laboratory test data. In addition, in situ analysis of paint on the surface of the vessel was not possible due to both the non-uniformity of multiple layers of paint and to the possible interference from the metal substrate with the analysis.



Although there is still no commercially available technology to analyze for metals in paint while it is still on the surface of the vessel (in situ), portable or hand-held X-Ray Fluorescence (XRF) analyzers, capable of measuring Pb, Cr, Cd and other metals in powdered paint at levels below 50 ppm, are now available. These analyzers can be used onsite (ship-side) and will provide results within 3 minutes after on-site sample preparation (scraping and grinding paint and transfer to sample cell). The ship-side analysis of the paint samples immediately after they are scraped and the subsequent immediate availability of the test results for scheduling of production activities would achieve "non-stop execution of the critical path".

#### PROBLEM

Testing of paint on ship surfaces is required prior to initiating other repair activities to identify levels of specific hazardous metals, such as lead (Pb), chromium (Cr), and cadmium (Cd) that may be present. The data are used to ascertain the specific environmental controls and personal protection equipment (PPE) that must be utilized during the repair work in order to ensure worker safety as required by Occupational, Safety and Health Administration (OSHA) regulations. Testing of up to 500 samples per carrier and 200 samples per submarine may be required. Production schedules and worker safety are dependent on these results.

Because sampling must be done and test results must be finalized before other repair/ production work can begin, ship repair/ production activities are often delayed. Turnaround time for laboratory testing, data review and approval of the samples can take up to 2 weeks. Shipyard personnel from production, engineering and quality assurance often work together using back and forth communication to designate and expedite specific sample testing and to modify testing sequences in order to get the "next needed" test results; while this approach is working (that is, gets the job done) in the shipyards, it is inefficient and requires significant oversight and interruption of normal work flows. Sample custody and tracking is time consuming and cumbersome. Ship-side analysis of the paint samples to obtain the required safety data on an immediate, as needed basis, is desired to eliminate costly delays in the scheduling and initiation of repair work and can result in the "non-stop execution of a critical path."

#### Continued from Page 3

#### PROJECT

In a project funded through Penn State's Applied Research Laboratory (ARL) via a U.S. Navy Repair Technology (RepTech) program, a portable COTS X-Ray Fluorescence (XRF) analyzer was evaluated, validated and demonstrated at ARL and at Norfolk Naval Shipyard (NNSY) for the ship-side analysis of heavy metals in paint. The analyzer used for this project was a Niton® XL3t GOLDD+™ XRF analyzer, loaned and rented from Thermo Scientific<sup>®</sup>. Additional vendors, including Oxford Instruments America, Inc. and Olympus NDT, Inc., also sell high-end portable XRF analyzers that have very similar product specifications and capabilities that are expected to provide similar results. Figure 1 shows the analyzer as seen in product website advertising on the left and as installed at ARL in a bench-top test stand, with protective lead cover and a laptop computer for remote operation.

Perhaps the most important task to ensure the success and eventual implementation of this project was to collect information from relevant shipyard functional areas regarding their needs for paint testing, their current processes and procedures, their concerns, their safety regulations and their quality assurance needs. Several teleconferences and one-on-one phone discussions were held with shipyard personnel from all four Navy shipyards representing the paint shops, engineering, quality assurance (QA), occupational safety



and health and environment (OSHE). Norfolk Naval shipyard was the lead yard for the project.

The information collected from these discussions was fundamental in establishing the following Objectives, Goals and Metrics (OGM) for the project:

- No radiation source; X-ray tube source only
- Accuracy Accurate analysis of Pb, Cr, and Cd in paint to levels required by OSHE
- Sensitivity Limit of detection for Pb, Cr, and Cd is less than or equal to 10% of OSHE requirement
- Comparability
  - XRF test results comparable to lab results on same samples
  - No false negatives for samples having >50 ppm of Pb, Cr or Cd
  - XRF test results on ELPAT proficiency samples are within required limits for lead
- Meet radiation safety requirements for use in industrial shipyard
- Meet production personnel needs for use in industrial shipyard environment
- Reduced time for availability of paint test results for work scheduling

The requirements for eight of these nine parameters were met and a short-path plan was established to quickly establish and demonstrate the ninth parameter. Each of these OGMs or parameters will be discussed briefly in the following discussion. Complete



Figure 1. The handheld COTS XRF instrument used in the Heavy-Metal Paint Contamination project. The instrument is shown on the left, with the inset showing the built-in data display. The photo on the right shows the instrument mounted in its bench-top stand and connected to the laptop computer used to record and analyze the data.

details were published in the final project report (Keay, 2012).

The selected XLt3 XRF analyzer contains an X-ray source and does not contain a radioactive source and thus meets the first shipyard requirement. The X-ray source used to generate the X-rays for analysis does present safety issues regarding potential exposure to X-rays of users and personnel in close proximity to the analyzer. However, shipyard regulations and procedures have already been established at all four Naval shipyards to allow for the safe use of X-ray generating analyzers in the shipyards. For this application, users would be properly trained following the established procedures and the analyzer would be used in a lead-enshrouded test stand with remote initiation of analysis. Thus the XRF analyzer can be used within the safety requirements of the shipyards.

The linearity, accuracy and sensitivity of the XLt3 SRF analyzer for lead, chromium and cadmium was documented at ARL using commercially prepared reference materials, such as NIST lead paint standard 2581. The NIST reference material and several other commercially prepared soil and paint reference materials were tested using the Mining mode test algorithm available in the analyzer. This algorithm is specifically designed to test low levels of metals in powdered matrices with organic background compositions similar to paint. Plots of the certified values for these reference materials vs. the measure XRF Mining mode test results are shown in Figure 2.

The linearity correlation coefficient for lead was found to be 0.9961 indicating that the response for lead is very linear up to about 4,500 ppm. The slope of the least squares regression analysis was 1.0258; a slope of 1.0 would indicate a perfect correlation of 1:1 (perfectly accurate) for XRF response vs. actual (certified) concentration. Thus the lead XRF response in the Mining mode is both linear and accurate. The limit of detection for lead was found to be 10 ppm.

The linearity correlation coefficients and slopes of the least squares regressions for chromium and cadmium similarly demonstrate their linearity and accuracy using the XRF Mining



Figure 2. These graphs demonstrate the XRF instrument's nearly linear behavior with respect to varying concentrations of three heavy metals when the convenient sample preparation technique developed under this project is used. The table shows some of the data measured; "<LOD" means that no signal was detected that exceeded the limit of detection (LOD).

mode, as show in the plots below. The limits of detection for chromium and cadmium were 20 ppm.

Norfolk Naval Shipyard requested that some of their Environmental Lead Proficiency Analytical Testing (ELPAT) lead proficiency samples be tested using the XRF Mining mode to ensure that the portable XRF analyzer could meet the certification requirements of the American Industrial Hygiene Association (AIHA) lead testing program. ELPAT samples are provided on a quarterly basis to Norfolk Naval shipyard and other laboratories around the country by the (AIHA) for laboratory certification.

Twenty-three ELPAT lead proficiency samples, ranging in concentration from 0.041 to 4.472 mg/m<sup>3</sup> (or 410 to 44,720 ppm), were analyzed "as is", without additional sample preparation, to determine whether the XRF analyzer with the Mining algorithm would pass the same ELPAT certification requirements that the laboratory must meet.

The ELPAT XRF test results data were plotted against the ASTM Reference Values in Figure 3. The XRF data correlate very well to the expected values as can be seen by the linearity correlation coefficient of 0.9968 and the equation of the line that nearly passes through zero. As well as confirming that the XRF analysis easily meets the requirements for the AIHA ELPAT samples, the ELPAT sample data also reconfirm the linearity and accuracy of the XRF Mining method for lead over two orders of magnitude. The ELPAT samples do not provide any data for the analysis of chromium or cadmium.

The above testing was done using certified and well-characterized, reference materials. Comparability of XRF results to shipyard laboratory test results on actual shipyard paint samples was another OGM established for this project, including no false negatives and comparability of XRF to Inductively Coupled Plasma (ICP) laboratory results to within +/-20%.

There were no false negatives found for samples having >50 ppm of Pb, Cr or Cd, when 30 shipyard paint samples were tested.



Figure 3. This graph shows that XRF data on lead contamination in paint collected by the portable instrument correlate closely with known values in the NNSY ELPAT XRF reference samples.

#### **Continued from Page 5**

However, the OGM for comparability of XRF to ICP laboratory results to within +/-20% was not met. With the sample preparation procedure that was used to prepare the shipyard paint samples for analysis, the XRF test results were biased high relative to the shipyard ICP laboratory results. This is shown in the figure 4. The average differences seen, XRF relative to ICP, for the lead analysis (+200% at the shipyard) and for chromium (+120% at the shipyard) do not meet the stated OGM comparability goal of +/-20% for samples containing more than 100 ppm of lead or chromium.

The high bias seen for the shipyard paint samples is most likely due to the sample preparation used (60 seconds grinding in a coffee grinder) and the relatively large particle sizes, particle size ranges and non-homogeneity that resulted. The use of a better grinding method in order to get the particle sizes down to less than 100  $\mu$ m (preferably 60  $\mu$ m), together with thorough mixing and transfer of a representative sample to the sample cell will be necessary to eliminate the high bias (*McEachern*).

The first step to implementing portable XRF analysis in the shipyard will necessarily be to finalize the sample grinding and preparation procedure. The shipyard will need to identify grinders appropriate for easily grinding small samples to the correct particle size and that would require no (or minimal) cleanup. The grinders should then be brought into the laboratory for demonstration and for comparison of the XRF and ICP test results on the resultant ground samples.

#### SUMMARY

This project has validated and demonstrated that portable XRF analysis of lead, chromium, and cadmium in paint meets the shipyard specified and required needs for safety, for analytical accuracy and sensitivity, for improved on-demand reporting of results and for practical adaptation to use in the shipyard environment. Eight of the nine project OGMs were met and a short-path plan to meet the ninth goal was outlined.

Discussions between shipyard managers at



Figure 4. This chart shows that XRF analyses based on data from the portable instrument fail to correlate with laboratory ICP-based analyses within  $^+$ - 20%. We believe this shortcoming will prove to be addressable by refining the sample preparation technique.

the final shipyard demonstration indicated that there is serious interest in implementing the XRF analysis of paint in the shipyard. At that demonstration, shipyard management began discussing ways to do the work needed to finalize the sample preparation procedure and then to move toward implementation.

#### **OTHER APPLICATIONS**

X-ray fluorescence is used to detect and, in some cases, quantitate metals primarily with molecular weights from 17 (chlorine) to 92 (uranium). A few lighter elements such as magnesium, aluminum and sulfur can also be measured, but the analysis itself requires helium blanketing or that it be done under vacuum. Organic analysis (compounds containing primarily carbon, hydrogen, nitrogen and oxygen) cannot be tested using XRF; only the heavier metals contained in an organic material can be detected. An application of this is the detection of lead in plastic toys.

Portable XRF analyzers are used to analyze for metals in environmental samples such as soils and dusts, and for testing lead in paint on bridges, old buildings and homes. A typical application that is already in use in some shipyards if for the positive alloy identification of incoming components; some XRF analyzers have data libraries that can identify up to 400 different alloys by measuring the % composition of each metal in the alloy. XRF is also used in scrap metal sorting, and for detecting the metallic components of welds or coatings; depending on the application, the sample matrix and the level of sample preparation, XRF may be able to detect the presence of certain metals, but may not be able to accurately quantitate the composition.

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McEachern, Gary, Thermo Scientific. Telephone conversation with Janice Keay, November 17, 2012.

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# **INSTITUTE NOTES**



iMAST's systems and operations division head, Ed Crow, points out various combat vehicles on loan to ARL Penn State to Dr. Burrow (right) and Mr. Smerchansky (left). The military vehicles support various projects currently underway at ARL Penn State, in support of Marine Corps efforts.



CIMP 3-D attendees tour facilities as part of a national additive manufacturing conference open to invited members of industry and government.



ARL Penn State's Director, Dr. Edward Liszka, discusses iMAST and other ARL activities with Vice Admiral Kendall Card, USN (Deputy Chief of Naval Operations for Information Dominance/Director of Naval Intelligence.)

#### Marine Corps Systems Command Visit

Senior members of the Marine Corps Systems Command (MCSC) journeyed to University Park recently to visit iMAST as part of a capabilities overview. Dr. John Burrow (Executive Director) and Mr. Jim Smerchansky (Deputy Commander, Systems Engineering, Interoperability, Architectures and Technology) headed up the group of visitors.

The Marine Corps Systems Command is the Commandant of the Marine Corps's agent for acquisition and sustainment of systems and equipment used to accomplish their warfighting mission. The command outfits United States Marines with literally everything they drive, shoot and wear. Their focus is the young Marine in harm's way, protecting him or her, and providing this warfighter the wherewithal to execute the mission. The command's team of professional civilian servants and active duty Marines equips the warfighter to win. They listen, learn, research, develop, test, procure and sustain whatever it takes to get Marines what they need, when they need it efficiently and for the best value possible.

#### Manufacturing Goes Digital

Penn State demonstrated cutting edge manufacturing technology recently when it hosted a Technology Showcase and Technology Exchange on additive manufacturing at its new Center for Innovative Materials Processing through Direct Digital Deposition (CIMP 3-D). The Technology Showcase, which was co-sponsored by the National Additive Manufacturing Innovation Institute, was used to acquaint industry and government organizations on the potential for additive manufacturing to reinvigorate the nation's industrial base. The Technology Exchange, which was sponsored by the Defense Advanced Research Projects Agency, involved in-depth technical discussions on critical enabling technology for wide-scale adoption of additive manufacturing issues important to the services at a time when breakthrough technologies are critical in producing more affordable components and systems that enhance performance and readiness, as America moves forward in the 21<sup>st</sup> Century. For more information about the CIMP 3-D program effort, contact Rich Martukanitz, Ph.D. at (814) 863-7282, or by e-mail at rxm44@psu.edu. You may also find additional information at the following web site: http://www.arl.psu.edu/mm\_lp\_cimp3d.php

#### Navy League Sea-Air-Space Expo

The 2013 Navy League Sea-Air-Space Expo is now in the books. This annual event provided an excellent opportunity for iMAST to continue acquainting senior members of the DoD, U.S. Navy, and the Marine Corps with our wide-range of activities. This year's theme, Maritime Crossroads: Strategies for Action, provided an excellent forum for learning about the various issues within the fleet. It also facilitated sharing further activities iMAST has on-going within ONR's Navy ManTech program effort within ARL Penn State. Keynote speakers included CNO, Admiral Jonathan Greenert, as well as the Commandant of the Marine Corps, General James Amos USMC. ARL Penn State's booth drew numerous distinguished visitors who viewed the full-scale improved lightweight shipboard watertight door, as well as other Navy ManTech project artifacts displayed at the event. The Navy League Sea-Air-Space event continues to provide an excellent venue for keeping up with new issues facing the naval services, to include the U.S. Coast Guard. Mark your calendars for next year's event which will be held 7–9 April 2014 at National Harbor, Maryland.



Reminder: Our 2012 annual report is on-line. Please visit: http://www.arl.psu.edu/documents/iMAST/12\_ annual\_rprt.pdf



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"We have to be careful that we don't break or harm the industrial base that we rely on for the long term. In our shipbuilding plan, the Navy has made a very concerted effort to stabilize requirements, drive affordability, and also balance consideration of our industrial base."

-Sean Stackley, Assistant Secretary of the Navy for Research, Development and Acquisition

## **CALENDAR** of **EVENTS**

2013

American Helicopter Society 69th Annual Forum		** Phoenix, AZ
Johnstown Showcase for Commerce		** Johnstown, PA
Letterkenny Showcase for Commerce	CANCELLED	Chambersburg, PA
Mega-Rust: Naval Corrosion Conference		Newport News, VA
Military Vehicle Exhibition & Conference		Detroit, MI
Expeditionary Warfare Conference		Panama City, FL
Fleet Maintenance & Modernization Symposium		** San Diego, CA
Modern Day Marine Expo		** Quantico, VA
Logistics Officers Association Conference	CANCELLED	** Dallas, TX
AUSA Expo		Washington, D.C.
DoD Maintenance Conference	CANCELLED	** Long Beach, CA
Defense Manufacturing Conference		** Orlando, FL
	American Helicopter Society 69th Annual ForumJohnstown Showcase for CommerceLetterkenny Showcase for CommerceMega-Rust: Naval Corrosion ConferenceMilitary Vehicle Exhibition & ConferenceExpeditionary Warfare ConferenceFleet Maintenance & Modernization SymposiumModern Day Marine ExpoLogistics Officers Association ConferenceAUSA ExpoDoD Maintenance ConferenceDefense Manufacturing Conference	American Helicopter Society 69th Annual ForumJohnstown Showcase for CommerceLetterkenny Showcase for CommerceMega-Rust: Naval Corrosion ConferenceMilitary Vehicle Exhibition & ConferenceExpeditionary Warfare ConferenceFleet Maintenance & Modernization SymposiumModern Day Marine ExpoLogistics Officers Association ConferenceAUSA ExpoDoD Maintenance ConferenceCANCELLEDDefense Manufacturing Conference

## 2014

14–16 Jan	Surface Navy Association Symposium	** Crystal City, VA
7–9 Apr	Navy League Sea-Air-Space Expo	** National Harbor, MD