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The Actinide Research Quarterly

of the Nuclear Materials Technology Division

Chief Scientist's Notes: Going Back to the Basics

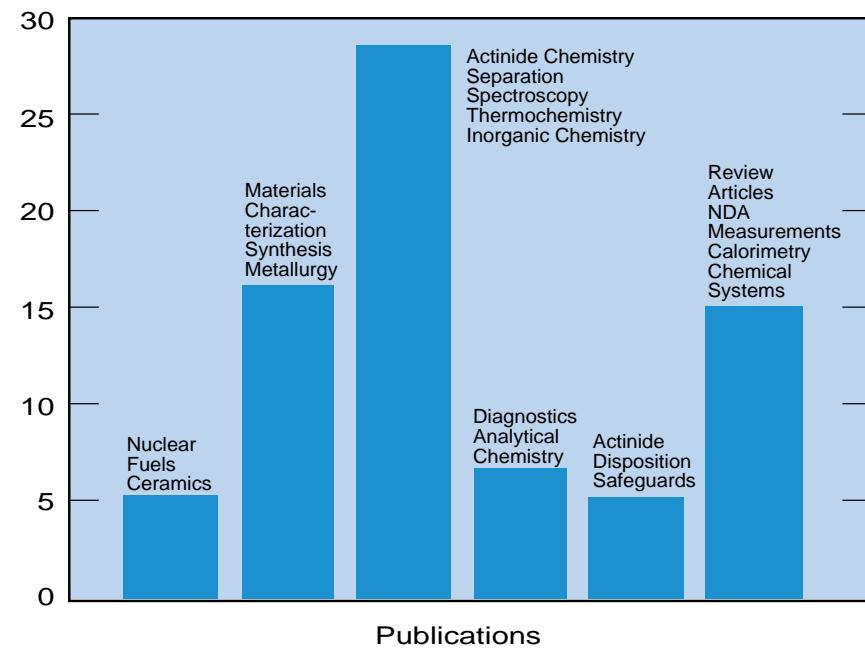
The Science and Technology Panel of the University of California President's Council on the National Laboratories has the primary responsibility for assessing the quality of the science and technology (S&T) programs of this Laboratory. As a partial fulfillment of this responsibility, in October 1994, the Nuclear Materials Technology (NMT) Division had a very successful division review done by the Laboratory Director-appointed External Review Committee. The Committee highly praised NMT Division members' scientific and technical accomplishments during the review year. The Laboratory's goals include such phrases as "a world-class laboratory...where science makes a difference," and "science serving society." Now, The Secretary of Energy Task Force on Alternative Futures for the Department of Energy National Laboratories (The Galvin Committee) notes "the laboratories' research role is a part of an essential, fundamental cornerstone for continuing leadership by the United States." Clearly, the future emphasis will be on going back to the basics of doing science.

When it comes to assessing scientific productivity, the most commonly applied metrics include patents filed, inventions disclosed, the quantity and quality of research papers published, citations received, and lists of technical problems solved. These metrics were heavily ascribed to all of the Laboratory S&T assessment efforts. Publication in

"...the laboratories' research role is part of an essential, fundamental cornerstone for continuing leadership by the United States."

Galvin Committee

Figure 1.
Publications by NMT Members (1993–1994).





**NMT Chief
Scientist K.C. Kim**

Until the development of the hydride-dehydride recycle process, the United States had no easy way of recovering plutonium from its nuclear weapons without generating copious amounts of mixed hazardous waste.

Going Back to the Basics (*continued*)

Budget shortfalls curtailed many essential programmatic activities.

It is heartening to see that a steady stream of technical reports has been processed through the division office even during this difficult time. As Division Director Bruce Matthews stated on one occasion, "It is essential that we focus on doing the actual science and technology committed to sponsors... Success in delivery will breed success in attracting out-year (immediate future) support."

The goal of the Actinide Research Quarterly is to communicate our scientific and technical progress among ourselves and to the outside community in general. The Quarterly is here for you to use by submitting your ideas, success stories, technical articles, and other accomplishments. Let this be your vehicle for helping us to go back to the basics of doing good science.

K.C. Kim

Hydride-Dehydride Recycle Process for Plutonium Components

Recent development of a hydride-dehydride recycle process provides a breakthrough plutonium-processing technology for the dismantlement and destruction of nuclear weapons. Past plutonium reprocessing technologies generated copious amounts of mixed hazardous waste that were difficult to dispose of. These past methods are no longer permitted, available, or acceptable for the removal of plutonium from nuclear weapons retired from the stockpile. Indeed, it is currently irresponsible to generate mixed hazardous waste because no repository exists for its disposal. As such, until the development of the hydride-dehydride recycle process, the United States had no easy way to recover plutonium from its nuclear weapons **without** generating copious amounts of mixed hazardous waste.

The hydride-dehydride recycle process developed at Los Alamos is an environmentally benign process that reduces hazards to workers and the environment and eliminates the generation of mixed hazardous wastes. This recovery process takes advantage of the fact that plutonium reacts easily with hydrogen gas. The principal advantage is the simple, fast, and reversible chemical reaction. Plutonium forms a metal hydride compound by reacting with hydrogen gas thousand of times faster than other metals do. The metal hydride then dissociates readily back to hydrogen and plutonium when heated. The use of hydrogen thus provides a convenient method for separating Pu from other materials within weapon components.

This recovery process is initiated by placing a plutonium-containing weapon component in the upper portion of the vacuum chamber (cold zone) as shown in Figure 2. An attached furnace tube and crucible with a resistive heating element (hot zone) is located directly below the nuclear component. A small amount of hydrogen gas supplied by a commercially available uranium hydride (UH₃) storage bed is introduced into the evacuated reaction chamber. The hydrogen gas reacts with plutonium from the weapon component to form a plutonium hydride compound (hydriding reaction). This hydride subsequently falls from the component and into the hot crucible where the heat initiates the release of hydrogen gas (dehydriding reaction). Hydrogen gas released from the hot zone is recycled to hydride more of the plutonium located in the cold zone. The hydriding-dehydriding continues between the cold and hot zones until the plutonium is completely extracted from the weapon component.

Melting and subsequent cooling of the plutonium powder produce a solid plutonium metal product that is ready for storage. During the plutonium melting process the hydrogen gas is pumped from the reaction chamber by a uranium powder storage bed. This hydrogen gas is stored at low pressure in the uranium bed and is readily available for future processing of other weapon components.

The whole process occurs well below atmospheric pressure and in the absence of any oxidants, which mitigates any possible explosion hazard of hydrogen gas. In addition the correct amount of hydrogen needed for the recycling reaction is easily and safely acquired by raising the temperature of the uranium hydride bed until the desired quantity of gas is obtained. This innovative method of hydrogen reagent supply eliminates the hazard of handling high-pressure gas cylinders within a plutonium facility. The reabsorption of the hydrogen gas by the uranium powder further enhances the safety of this process and prevents hydrogen gas from being released to the environment.

This process enables a key step in the verification and dismantlement of the world's nuclear weapon stockpile. Currently, without this technology, the United States has no permissible means of dismantling nuclear weapons and removing plutonium for subsequent bilateral or international inspection and storage. This process is the principal technology for warhead dismantlement and thus contributes to reducing the global nuclear danger. It may be applied to any suggested scenario that requires plutonium retrieval, including vitrification, reactor burning, or geologic storage, before final plutonium disposition. The process itself produces a minimum of waste and mitigates numerous environmental hazards formerly associated with weapon dismantlement operations. This technology eliminates mixed hazardous waste streams for which there is no current U. S. repository.

The hydride-dehydride process has other applications as well. It can be used immediately to clean up plutonium metal from manufacturing equipment such as casting molds, tantalum and ceramic crucibles, ceramic and metal stirrers, and steel furnace tubes. A prototype hydride-dehydride recycle system is currently being used on a production scale to support several LANL programs. These programs include the Surveillance Program (study of aging effects within the nuclear package of a weapon), Fire Resistant Pit Program (develop fire-resistant container for the nuclear package), Waste Minimization Program (develop waste-free plutonium processing technologies) and finally Automated Recovery Integrated Extraction System Program (dismantlement program).

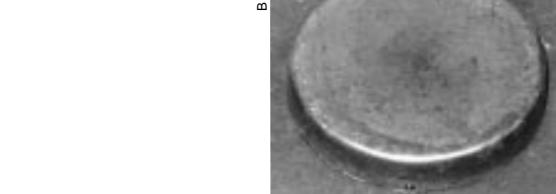
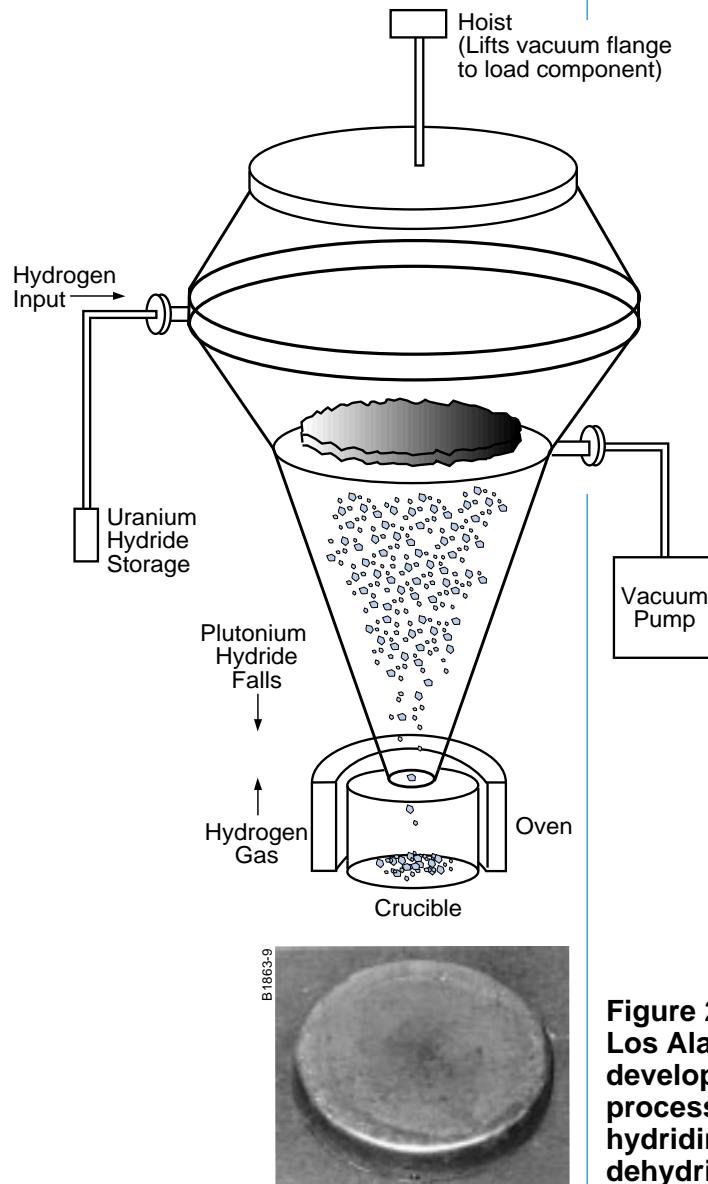


Figure 2. The Los Alamos-developed process for hydriding and dehydriding of nuclear weapon parts for plutonium recovery. It also shows the final product.

Bart F. Flamm and Garlan M. Isom are the principal developers of this process.

Electrolytic Decontamination of Oralloy

Throughout the Department of Energy (DOE) Complex, there are holdings of oralloy (highly enriched uranium) contaminated with plutonium (Pu) and americium (Am). Contaminated oralloy cannot be sent to Oak Ridge, the nation's oralloy disposition site,

until its surface is cleaned to a swipable alpha activity level of Pu and Am below 20 disintegrations per minute (dpm)/100 cm². Together, the NMT and Engineering Sciences and Applications (ESA) Divisions from Los Alamos National Laboratory have demonstrated the electrolytic method of decontaminating oralloy. This method results in > 99.9% reduction in the waste stream compared to previously used methods. Additionally, the electrolytic decontamination method has been used successfully to clean other metals.

In the past cleaning to this extremely low level of Pu and Am was accomplished with difficulty using a method that required large quantities of hot, concentrated nitric acid in an acid spray leach process. Rocky Flats Environmental Technology Site (RFETS), realizing that they had large holdings of contaminated oralloy, tried to develop a new method of cleaning oralloy and initiated the concept of electrolytic decontamination of oralloy. Unfortunately, before this method of cleaning could be demonstrated, operations at RFETS were halted. Thus, in the spring of 1992 RFETS and NMT began negotiations to demonstrate electrolytic decontamination at the Los Alamos Plutonium Facility. After a few months of this cooperative arrangement, NMT continued this effort on its own. Part of the interest for the Laboratory was to find a method of decontaminating oralloy for the ARIES (Automated Retirement and Integrated Extraction System) project, as well as other stockpile support activities.

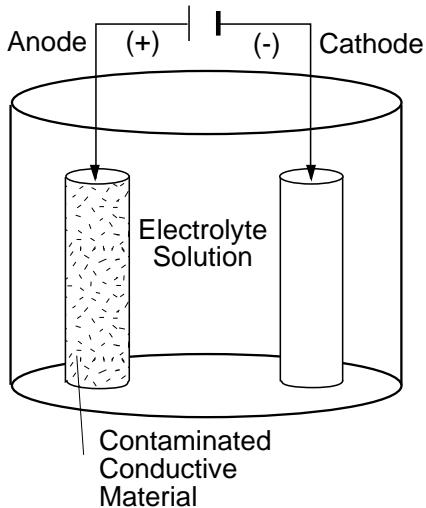


Figure 3. Under electric current flow, the contaminant (Pu) is removed from the surface of the anode and goes into the electrolyte solution. When the current is turned off, the contaminant eventually precipitates out.

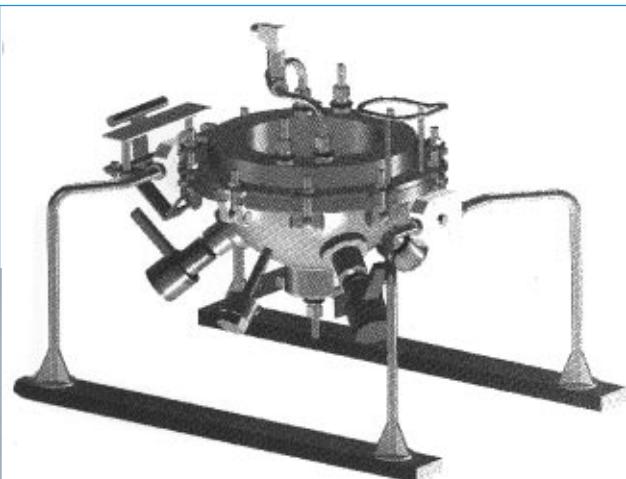
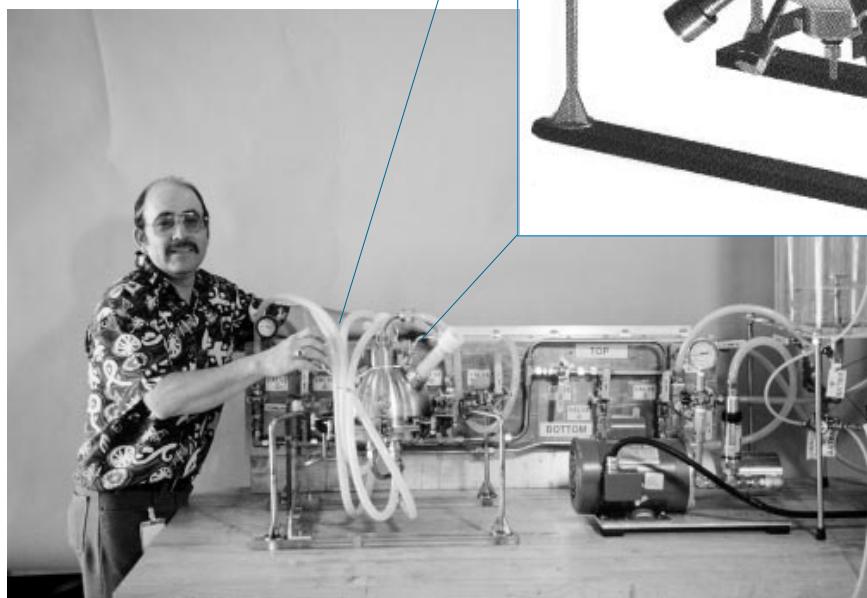


Figure 4. The electrolytic decontamination apparatus. The process can be used on a wide variety of conductive metals including stainless steel.

Electrolytic decontamination is similar to the common industrial practice of electro-polishing and is accomplished by applying a low DC voltage through an electrolyte to induce a chemical reaction. Contamination is removed at the anode, the working electrode, in this case the oralloy, and goes into solution (Figure 3). The cathode, or counter electrode, can be a variety of materials, but we typically use stainless steel. Because of their capacity to handle nitrates, RFETS planned to use sodium nitrate as the electrolyte. In the presence of sodium nitrate the contaminants form a precipitate. This precipitate formation leads to easy separation of the waste from the solution; thus, the electrolyte solution can be recycled, and waste is greatly reduced compared to the acid spray leach process.

Results of early beaker experiments with contaminated oralloy coupons demonstrated the effectiveness of the electrolytic technique, reducing swipable alpha levels from $> 1,000,000$ dpm to < 2 dpm. (In the early phases of this project, we were unable to distinguish between U, Pu, or Am alpha; thus, we removed all of the swipable surface material.) Additionally, it was during these coupon experiments that it became evident that the precipitate would form and that the same electrolyte could be used for numerous coupon tests. Also, subsequent analysis of the electrolyte revealed no measurable amount of contamination in solution so that the electrolyte can be discarded as industrial waste.

These very successful experiments launched the testing of a fixture to hold hemishells for decontamination (Figure 4). The fixture was designed to follow the contour of the parts to be decontaminated so as to clean the surface uniformly and to reduce the necessary volume of electrolyte solution as well. Also, we were fortunate to get a clean glovebox to put the fixture in for testing, in which typical inside swipes are 20 dpm or less. These hemishell experiments gave similar results to the earlier coupon tests, starting with swipable values much greater than $1,000,000$ dpm/ 100 cm^2 and ending with swipable values < 20 dpm/ 100 cm^2 .

Realizing that our swipe measurements were for total alpha, we began work with the Chemical Science and Technology (CST) Division to investigate the discrimination of the different alpha particle energies. During these initial tests, we reran some of the swipes from the previous tests and obtained values as low as 8 dpm/ 100 cm^2 Pu and Am. Distinguishing between the alpha activity levels of the oralloy and the alpha activity of the contaminants is important. It prevents overcleaning, thus it enables the cleaning technique to run for shorter times, resulting in less solid-waste product generated and lower exposures to personnel. Because of this effort, we now have an alpha spectrometer in Building PF-4, Room 106, that we use to identify the U, Pu, and Am alpha particles. Also, we have begun development on an alpha spectrometer to determine fixed (unswipable) contamination on the oralloy hemishell. This latter effort will be the development of a completely new instrument not currently available commercially.

The third-generation fixture has recently been installed in PF-4 and has been fully tested by decontaminating a number of oralloy parts. The decontaminated parts will be shipped to Oak Ridge for disposition when the Y-12 facility is ready to receive them. Moreover, other projects using electrolytic decontamination have begun, such as using *in situ* electrolytic decontamination of gloveboxes and working with Sandia on an automated process to clean cans to a level at which they can be removed safely from the glovebox system.

Because of the success of the electrolytic process, it is currently the base line oralloy decontamination technology for stockpile support activities. As a result of our success with electrolytic decontamination of oralloy, we have drawn national recognition—including a request to DOE from Lawrence Livermore National Laboratory that Los Alamos National Laboratory be the “center for oralloy decontamination” for the nation.

Contributors to the project are **George Campbell, Malcolm Fowler, Mary Esther Huerta, Lorenzo Jaramillo, Lonnie Morgan, Ed Martinez, Tim Nelson** (project leader), **John Parker, Wilfred Romero, Wayne Smith, Nelson Stalnaker, Len Stapf, and Lee Vikdal.**

This method results in >99.9% reduction in the waste stream compared to previously used methods. Because of the success of the electrolytic process, it is currently the base line oralloy decontamination technology for stockpile support activities.

NMT Team Profile

This team helps to fulfill the Laboratory's mission to reduce the nuclear danger by researching issues attendant to maintaining the downsized nuclear weapons stockpile: its safety, security, and reliability.

Team members are Team Leader Joe Baiardo, WRD&T Project Leader John Haschke, Tom Allen, Barbara Cort, Larry Cox, Bart Flamm, Marty Reisfeld, John Ward, Joel Williams, Paul Watson, Chuck Radosevich, Fidel Vigil, Gary Isom, Bob Pruner, Ben Jacquez, and Trish Wright. Former team member Joe Martz is Group Leader of NMT-5.

Applied Weapons Research and Development— A Work Profile

The Applied Weapons Research and Development (R&D) Team, NMT-5, (Weapons Component Technology Group) is involved in research to improve the fabrication, performance, and surveillance of plutonium components within nuclear weapons systems. The six staff members, six technicians, and one postdoctoral fellow who comprise this team help fulfill the Laboratory's mission to reduce the nuclear danger by maintaining the downsized nuclear weapons stockpile and ensuring that it remains safe, secure, and reliable. The team also supports the Lab mission through research in weapons dismantlement, disposition of excess nuclear material, and remediation of nuclear waste.

A key concern of stockpile maintenance is stockpile aging. Plutonium weapons components will not remain functional forever. The question is, how and when will they fail? The assessment of the performance of aging weapons warrants a major effort of the Applied Weapons R&D Team to characterize fundamental and derived properties of plutonium metal and alloys as a function of their age. The goal is to identify, quantify, and model important property changes. Unlike chemical reactions, processes driven by radioactivity are not amenable to accelerated aging techniques. Fundamental processes that might alter the geometry or density of a component include chemical corrosion, ingrowth of decay products, and structural change of the metal. Studies characterize derived properties of new plutonium, define those properties for aged material, and determine how rapidly materials are changing with age. Fundamental property results must then be correlated with derived properties used in predictive calculations and compared to results from aboveground experiments. It is hoped that enhanced techniques for pit surveillance will ultimately result from this work.

Stockpile stewardship also involves the maintenance of a viable weapons manufacturing capability. Team members are involved in several projects with the aim of decreasing waste, increasing efficiency, and reducing operational hazards. For example, the team is currently designing and procuring a production-scale supercritical fluid cleaning system that uses and recycles nonhazardous carbon dioxide solvent to clean plutonium components before pit assembly to prevent corrosion during weapon storage and deployment. Reflectance Fourier transform infrared methods are under development for verifying component cleanliness.

Efforts to enhance the safety of nuclear weapons are actively pursued. For example, an aqueous nitrate technology for thermally applying thin coatings of erbium oxide to metals and other substrate materials is being developed for possible application in fire-resistant pits and in reusable crucibles and molds for manufacturing and recovery processes. Other studies identify and address safety concerns posed by reactive and pyrophoric materials during the maintenance and disassembly of weapons.

Dismantlement of retired weapons and recovery of plutonium are essential for reducing the nuclear danger. These efforts form the starting point for any proposed plutonium disposition strategy. Recent studies have demonstrated that a recycle hydride-dehydride concept is a safe, rapid and efficient method for separation (> 99.95%) of plutonium from other materials. The potential for using this technology to extract plutonium from U.S. and Russian weapons components has generated widespread interest, including reciprocal U.S./Russian tours of laboratory facilities. NMT Division has submitted this technology as an entry to the 1995 "R&D 100" competition. (See article this issue.)

Storing surplus nuclear material safely before disposition is yet another aspect of reducing the nuclear danger. The team recently conducted several technical assessments related to interim and long-term storage of plutonium. In addition to identifying material forms suitable for storage, they have evaluated problem situations with stored materials, contributed to a DOE assessment of storage issues and vulnerabilities, and played a key role in defining the DOE criteria for safe storage of metal and oxide. The scope of this activity has recently expanded because of increased interaction with the Laboratory's Russian counterparts. Research into promising technologies for storage continues as well. During the past year, substantial progress has been made in developing and evaluating acoustic resonance spectroscopy as a method for nondestructive surveillance of stored containers.

Lastly, the team is directly involved in waste minimization and remediation research, primarily through the development of a plasma-based decontamination process. The technology is under development in joint collaboration with the Chemical Science and Technology and Physics Divisions where fundamental aspects and potential applications of the process are being investigated.

Recent Publications and Reports

The following reports and publications were processed by the division office during this quarter:

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C. E. Olsen, "The Magnetic Susceptibility of Alpha Phase Plutonium Below 300 Kelvin and the Electronic Structure of Plutonium Metal," *Journal of Metals and Alloys*, in press, (1995).

G. D. Jarvinen and B. F. Smith, "Water-Soluble Chelating Polymers for Removal of Actinides from Waste Waters," Efficient Separations and Processing Integrated Program Technical Information Exchange Meeting; Gaithersburg, MD, January 24-26, 1995 Los Alamos National Laboratory document LAUR 95-0185.

N. G. Pope, R. E. Brown, and W. J. Turner, "TA-55 Facility Control System Upgrade - Facility Data Acquisition Interface System Functional and Operational Requirements," Los Alamos National Laboratory report LA 12914 (1995).

T. O. Nelson, M. E. Huerta, L. Jaramillo, A. N. Morgan III, H. E. Martinez, W. R. Romero, and L. H. Staaf, "Using Electrolytic Decontamination Methods for Pollution Prevention;" T. R. Mills, W. A. Punjak, L. D. Schulte, C. A. Smith, W. B. Smith, W. D. Smyth, S. B. Schreiber, and S. L. Yarbro, "Acid Recovery and Recycle"; J. Foropoulos, Jr., "A Low Temperature Gas-Solid Process for the Destruction of Chlorocarbons"; 1995 Los Alamos National Laboratory Pollution Prevention Showcase; Santa Fe, NM, January 16-18, (1995) Los Alamos National Laboratory documents LAUR 94-4401, 4402, 4013.

E. Garcia, "Distillation Separation of Chloride Salts from Plutonium," DOE Office of Technology Development for Technical Integration and Exchange Meeting; Washington, D.C., January 24-26, 1995 Los Alamos National Laboratory document LAUR 94-4267.

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P. K. Benicewicz, J. P. Roberts, and A. J. Taylor, "Scaling of Terahertz Radiation from Large Aperture Biased Photoconductors," *Journal of the Optical Society of America B*, **II**, 2533-2546 (1994).

M. H. West and K. M. Axler, "Thermodynamic Modeling of Hydrogen Fluoride Production Relevant to Actinide Residue Treatment," Los Alamos National Laboratory Report LA -12909-MS (February 1995).

P. C. Lopez, K. M. Axler, R. Edwards, W. J. Griego, and R. Pereyra, "Low Temperature Compatibility Testing of Plutonium with Selected Substances," 1995 TMS Annual Meeting and Exhibition; Las Vegas, NV, February 12-16, 1995 Los Alamos National Laboratory document LAUR 94-2746.

S. B. Schreiber, S. L. Yarbro, N. G. Pope, and R. S. Day, "Advanced Testing Line for Actinide Separations (ATLAS)," Los Alamos National Laboratory Environmental Stewardship Conference; Santa Fe, NM, February 6-8, 1995.

P. L. Wallace, M. H. Mueller, L. D. Calvert, and R. Jenkins, "The new ICDD Metals and Alloys Indexes: Usefulness and potentialities," *Powder Diffraction*, **9** (4), 239-245 (1994).

L. E. Cox and R. Martinez, "Short-range Atomic Structure of 1 wt. % Ga δ-stabilized plutonium by X-ray-absorption fine-structure spectroscopy," *Physical Review B*, **51**, No. 2) (1995).

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D. L. Clark, J. C. Gordon, J. C. Huffman, R. L. Vincent-Hollis, J. G. Watkin, and B. D. Zwick, "Preparation and X-ray Structures of K[Ln(O-2,6-i-Pr₂C₆H₃)₄] (Ln = La, Nd, Er). Extended Chain Structures of Lanthanide Tetrakis(Aryloxide) Anions Bridged by Potassium-η-Arene Interactions," *Inorganic Chemistry*, **33**, 5903 (1994).

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L. R. Avens, S. G. Bott, D. L. Clark, A. P. Sattelberger, J. G. Watkin, and B. D. Zwick, "A Convenient Entry Into Trivalent Actinide Chemistry: Synthesis and Characterization of AnI₃(THF)₄ and An[N(SiMe)₃]₂ (An = Np, Pu)," *Inorganic Chemistry* **33**, 2248 (1994).

Newsmakers

NMT R&D 100 Entries

Each year the Chicago-based *R&D Magazine* chooses the 100 most innovative inventions/technologies of the previous year for presentation of its prestigious international **R&D 100 Awards**. NMT Division registered a record three entries for the competition cycle this year. They are

- Wendel G. Brown's **Minimum-lubricant Asymmetric Motion Lathe** (MAC Lathe)
- **Noninvasive Chemical Concentration Analyzer**, by Douglas K. Veirs (NMT-6), Noah G. Pope (NMT-2), David Sanchez (NMT-6), and Vicente Sandoval (NMT-6)
- **Hydride-Dehydride Recycle Process**, by Bart F. Flamm (NMT-5), Joseph C. Martz (NMT-5), Garland M. Isom (NMT-5), and John M. Haschke (NMT-5) (see details in this issue)

New Ph.D.— James McHale

James M. McHale, a graduate research assistant in NMT-6, has completed his Ph.D. dissertation entitled "**Solution-based Synthesis of Perovskite-type Oxide Films and Powders**." McHale's dissertation was submitted to the Temple University Graduate Board. Nicholas Coppa, NMT-6 Staff Member, served as McHale's advisor for his dissertation work.

New LDRD/CD Proposal

The Laboratory-directed research and development (LDRD) STB Office informed the division in early January that a new LDRD/CD proposal, "Polymers for Nuclear Materials Processing," by **Gordon Jarvinen** (NMT-6) was selected for 1995 funding out of the LDRD reserve.

Featured Speaker

Barbara Cort (NMT-5) was a featured speaker on Science Career Day at Los Alamos Middle School, January 13, 1995.

CIC-9-d950029



Minimum-lubricant Asymmetric Motion Lathe

Los Alamos

NATIONAL LABORATORY

The *Actinide Research Quarterly* is published quarterly to highlight recent achievements and ongoing programs of the Nuclear Materials Technology Division. We welcome your suggestions and contributions.

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