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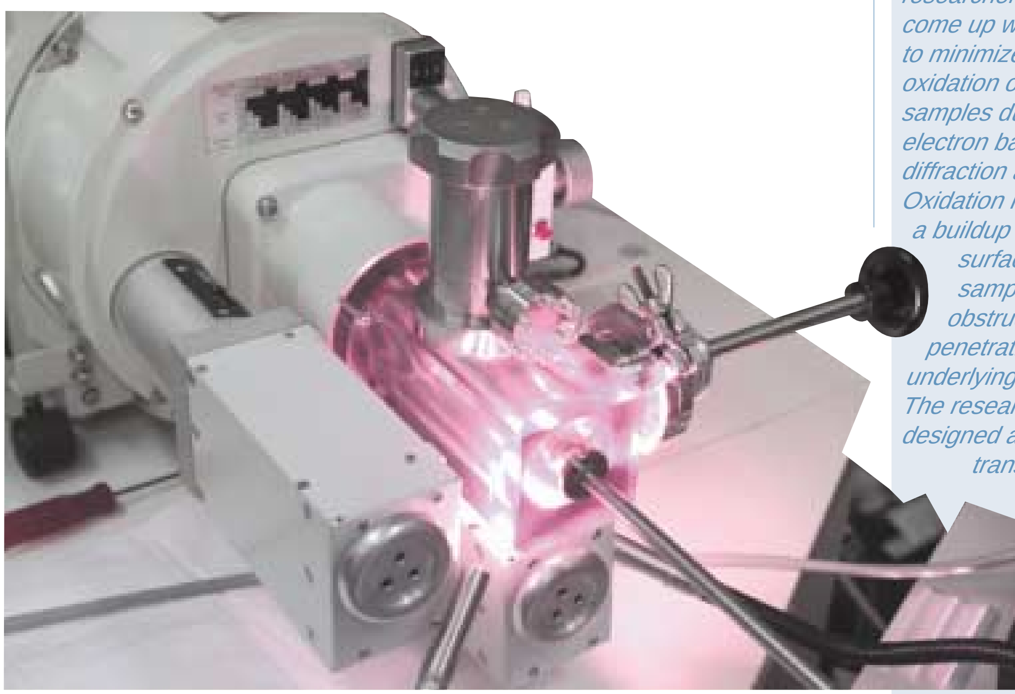
In This Issue

Electron Backscattered Kikuchi Patterns of a Plutonium Alloy Captured for the First Time

New Technique Minimizes Surface Oxidation, Allows for Better Electron Penetration to Underlying Metal



Los Alamos researchers have come up with a way to minimize surface oxidation on plutonium samples during electron backscattered diffraction analysis. Oxidation results in a buildup on the surface of the sample that obstructs electron penetration to the underlying metal. The researchers have designed a device to transfer the sample to the scanning electron microscope chamber under vacuum. The device includes an entry-port adapter (the adapter's polycarbonate block has been illuminated with pink light in this photo; it doesn't really glow) and the vacuum suitcase (the black cylinder sitting atop the entry-port adapter). The scanning electron microscope chamber is the large cylinder located behind the adapter.

Plutonium is one of the most complex elements known to science. It may experience more than six atomic structure changes, transforming itself from one crystallographic phase to another, and each phase has different properties. Understanding the phase transformations, and in particular the microstructural evolution and aging behavior of plutonium and its alloys, is important to maintaining the safety and reliability of the weapons in the nuclear stockpile.

The relative crystallographic orientation of separate grains within a material, also called crystallographic texture, has long been known to strongly influence material properties. Researchers have typically obtained bulk texture data through x-ray or neutron diffraction, while transmission electron microscopy has provided local texture information. Recent technological advances in microscopy techniques have provided a means to evaluate crystallographic texture using common

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Kikuchi Patterns

Contributors to this article are: **Carl Boehlert** and **Jeremy Mitchell** (NMT-16), and **Roland Schulze** (MST-6). Other researchers on the project are **Thomas Zocco** (NMT-5); and **Jeffrey Archuleta**, **Ramiro Pereyra**, and **Doug Farr** (NMT-16).

metallurgical preparation techniques. This development has been in the field of electron backscattered diffraction through the automated collection and indexing of electron backscattered Kikuchi patterns.

Electron backscattered diffraction analysis is ideally suited for plutonium investigations because of the complex phases and phase transformations possible. However, using electron backscattered diffraction to analyze plutonium has proved elusive for several reasons, one of which is rapid surface oxidation. This results in the buildup of an amorphous surface layer that acts as an obstacle for electron penetration to the underlying crystalline metal.

This view inside the scanning electron microscope chamber shows a sample (shown magnified in the inset) on a puck assembly. A phosphor screen (the disk-shaped object) and a charge-coupled device camera are shown to the right of the sample. When backscattered electrons bombard the phosphor screen, a network of Kikuchi bands is imaged by the camera.

A team of Los Alamos researchers has come up with a way to minimize surface oxidation when using electron backscattered diffraction to analyze plutonium. They have designed a device to transfer the sample to the scanning electron microscope chamber under vacuum. The device includes a vacuum suitcase and scanning electron microscope entry-port adapter.

The team's research shows that maintaining a low-oxygen environment after cleaning a sample's surface is important to successfully observing electron backscattered diffraction patterns. The new technique has allowed researchers for the first time to evaluate crystallographic texture through the automated collection and indexing of electron backscattered Kikuchi patterns.

When incident electrons penetrate a sample in electron backscattered diffraction, they may experience a variety of elastic scattering events until the sample absorbs their energy, or they may scatter elastically and escape from the specimen surface as backscattered electrons. The diffracted backscattered electrons are emitted in a series of bands, known as Kikuchi bands, which relate to the crystal planes and their orientation within the sample.

To capture Kikuchi patterns, a phosphor screen is placed in a scanning electron microscope vacuum chamber close to a steeply inclined sample. When the backscattered electrons bombard the phosphor screen, a network of Kikuchi bands is imaged by a camera. The positions of the bands, which contain information relating to the symmetry and orientation of the crystal, are identified.

It is thought that sharp Kikuchi patterns are formed from backscattered electrons that have experienced only elastic interaction (low-loss electrons). This indicates that electron backscattered diffraction is surface-sensitive and the technique and quality of the patterns is strongly dependent on sample preparation. This is especially true for high-density materials such as plutonium.

Researchers estimate that within a distance of nine nanometers below the surface of a plutonium sample, 95 percent of the incident electrons would experience a collision resulting in a permanent energy loss for the electron. This energy loss would result in lost structural information provided by the electron. Therefore, researchers expect that the bulk of the electron backscattered diffraction information for plutonium comes from only the top several nanometers of the surface.

The material used by the Los Alamos team in its initial evaluation was a plutonium-gallium alloy containing the face-centered-cubic atomic structure (the same structure that is predominately used in nuclear weapons). For most nonreactive metals, simple metallographic polishing followed by transfer through the air to the scanning electron microscope is sufficient for successful electron backscattered diffraction capture.

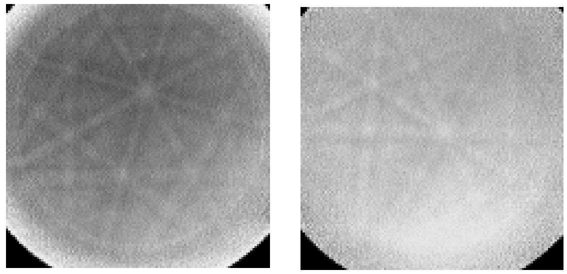
However, this isn't the case for highly reactive metals such as plutonium, and additional surface cleaning, such as ion etching—a method to remove the surface of a material by bombarding it with accelerated ions in a vacuum—is necessary.

For this reason, the research team used a multifunctional ultrahigh-vacuum Auger / energy loss spectroscopy (EELS) instrument to characterize and remove surface layers contaminated with chemical impurities, in particular carbon and oxygen. (For more information on the Auger / EELS instrument, see Actinide Research Quarterly, 4th Quarter, 2000.)

In addition to small plutonium peaks, researchers observed large carbon and oxygen peaks and a small fluorine peak. To remove the surface layers concentrated with contaminants, the sample surface was bombarded repeatedly by argon ions used in the ion-etching process. Along with removing the surface layers concentrated with oxygen and carbon, ion etching enhanced the grain boundary contrast (i.e., the grains were topographically distinct).

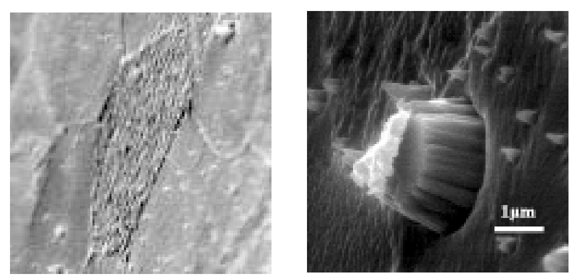
One of the most interesting aspects of the ion-etched microstructure was that the bulk impurities tended to etch at a different rate than the plutonium-gallium metal. This resulted in toothlike features that protruded up to two micrometers (two-millionths of a meter) above the surface. By minimizing ion etching to remove only those surface layers concentrated with carbon and oxygen, the surface protrusions, which interfere with the backscattered electrons used for diffraction, were likewise minimized.

Most environmentally sensitive materials that require surface cleaning can be transferred from the ion etcher through air to the scanning electron microscope vacuum chamber. For materials that oxidize rapidly, this jeopardizes surface integrity. For the plutonium-gallium sample, atmospheric transfer did not result in successful Kikuchi pattern acquisition, and this was attributed to the formation of a thick, amorphous surface oxide layer as a result of air exposure. Even an atmospheric exposure of only one second after ion etching led to a surface that did not exhibit Kikuchi patterns from the underlying plutonium-gallium metal.



These images show the electron backscattered Kikuchi patterns captured from two grains. The positions of the bands contain information relating to the symmetry and orientation of the crystals.

After the initial patterns were captured, researchers left the sample in the scanning electron microscope vacuum chamber for 68 hours and then successfully reanalyzed the sample for Kikuchi patterns. The surface oxide on the plutonium-gallium metal did not thicken enough within the vacuum environment to prevent researchers from observing the diffraction patterns, illustrating the minimal effect the vacuum environment has on surface oxidation.



These images obtained through scanning electron microscopy show low- and high-magnifications of an ion-etched microstructure. Bulk impurities on the surface of a sample result in toothlike protrusions (seen in the photo at right) that interfere with the backscattered electrons used for diffraction. By minimizing the ion etching to remove only those layers concentrated with carbon and oxygen, the surface protrusions were likewise minimized.

Researchers believe this new sample preparation and characterization technique will provide a powerful means to further understand phase-transformation behavior, aging behavior, and texture in the complicated plutonium and plutonium-alloy systems.■

Researchers Study Gases in Sealed Containers of Plutonium Oxide Materials

Shelf-Life Surveillance Project Will Help Answer Questions About Long-Term Storage of Waste

Contributors to this project are: **Tom Allen** (NMT-15); **Simone Balkey** (NMT-2); **John Berg, Max Martinez, Jim McFarlan, John Morris, Dennis Padilla, Karen Rau, Kirk Veirs,** and **Laura Worl** (NMT-11); **David Harradine** (C-PCS); and **Dallas Hill** and **Coyne Prenger** (ESA-EPE).

All photos by **Mick Greenbank** (NMT-16).

Los Alamos researchers are studying the behavior of oxide materials in sealed containers as part of a new Department of Energy (DOE) program aimed at safely storing large quantities of plutonium-bearing materials for up to 50 years.

Experience with pure plutonium oxide and with impure materials has shown that gases generated by catalytic or radiolytic processes may accumulate over time. Of concern to researchers are the generation of hydrogen gas from adsorbed water, the generation of water in the vapor phase at elevated temperatures, and the generation of hydrochloric or chlorine gases from the radiolysis of chloride-containing salts that exist as impurities in the oxides. The combination of chloride-bearing gases and condensed water may corrode a storage container.

Researchers have designed these instrumented small-scale (left) and large-scale shelf-life surveillance containers, which will be used to monitor gases over oxide materials.

Materials destined for long-term storage include metals and oxides that are stabilized and packaged according to a standard that requires a set of nested, welded stainless-steel storage containers. The recently revised standard, DOE-STD-3013, allows for a wider range of plutonium content (containing at least 30 weight percent plutonium plus uranium) in the stored materials. The earlier standard specified storage for plutonium-bearing metals and oxides to contain at least 50 weight percent plutonium plus uranium. The revised standard also specifies that only materials that are represented in Los Alamos' Material, Identification, and Surveillance (MIS) Program may be packaged for long-term storage.

The Los Alamos Shelf-Life Surveillance Project will monitor gases over oxide materials in a limited number of full-scale 3013 inner containers and in many small-scale containers. Oxide materials representing inventories destined for long-term storage throughout the DOE complex will be monitored.

The MIS Program has evaluated 33 items from Hanford and Rocky Flats Environmental Technology Site. Residuals from these evaluated items make up the surveillance items to date. Future materials will come from the Los Alamos inventory and from other sites.

The project includes researchers from Actinide Chemistry Research and Development (NMT-11), Actinide Processing (NMT-2), Physical Chemistry and Applied Spectroscopy (C-PCS), and Energy and Process Engineering (ESA-EPE), and it involves two parallel studies.

A small-scale study will monitor many 10-gram samples for short time periods. This study continues the work initiated by Tom Allen of Pit Disassembly and Nuclear Fuels Technologies (NMT-15). A large-scale study will monitor, for longer periods, plutonium oxides prepared according to the 3013 standard, oxides exposed to high-humidity atmospheres, and oxides containing chloride salt impurities.

The small samples will allow researchers to compile a database of many materials prepared according to various site-specific packaging methods. The large samples will give the precise behavior of a limited number of full-scale samples. Comparison between the two sample types will determine the degree of confidence in small-sample experiments and fundamental measurements in predicting the long-term behavior of real materials.

Researchers have designed instrumented storage containers that mimic the inner storage can specified in the 3013 standard at both full- and small-scale capacities (2.34 liters and 0.0045 liter, respectively). The containers are designed to maintain the volume-to-material mass ratio while allowing the gas composition and pressure to be monitored over time.

The full-scale cans are instrumented with a Raman fiber-optic probe, a gas chromatography / mass spectrometer sampling port, an acoustic resonance chamber, two corrosion monitors, and pressure and temperature sensors. Data collection for the full-scale containers is automated to reduce worker radiation exposure.

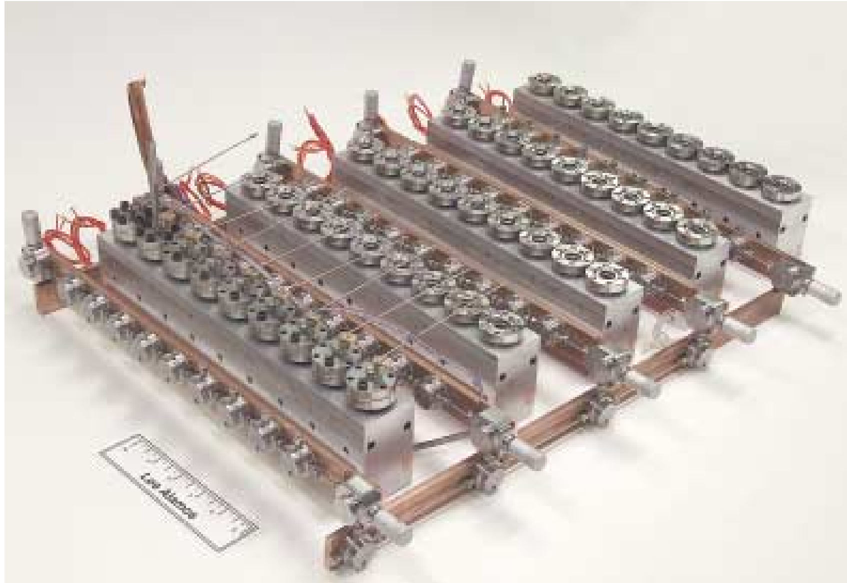
To minimize the effect of invasive gas sampling on the experiment, gas chromatography sampling will be limited. The majority of the gas compositional data for the large-scale study will be obtained noninvasively through Raman spectroscopy.

The small-scale containers are designed with a microliter gas-sampling capability, and pressure and temperature sensors. These small containers will be stored in a heated array to reproduce the increased temperatures arising from radioactive self-heating. The tubing between the small containers and the gas-analysis equipment will be heated to 100 degrees Celsius to avoid condensation of gases. Pressure and temperature data will monitored continuously and gas analysis will be conducted at periodic intervals.

An initial 45 samples are planned for the small-scale study, and will include experimental blanks and plutonium oxide samples from DOE sites. The samples are selected to address known existing uncertainties in gas generation and safety considerations associated with specific material types being considered for storage.

They also will contain samples of the actual materials going into the large-scale study. A percentage of the oxide materials in the small-scale study will be used for data validation of gas-generation models in development across the DOE complex.

In the large-scale study, 10 large-scale instrumented inner 3013 containers are being installed at Los Alamos' Plutonium Facility in Building PF-4 to study compositional changes in gases over plutonium oxide materials for an extended time. Nine of the containers will hold characterized oxide materials in varying forms obtained from NMT-2; the 10th container will be an experimental blank.



This small-scale array has 45 sample containers located in the heating block with the heated manifold. The nine containers in the left-hand row have the lids in place, but do not have the gas sampling transfer lines connected.

A rack system to hold the large array has been designed, fabricated, and tested. Like the array for the small containers, it contains a heated gas manifold to minimize the effect of gas condensation and to ensure quantitative gas chromatography analysis.

Technical issues that will be explored through the large-scale experiments include the effect of specific surface area, water, salts, organics, fill gas, and mixed oxides on gas generation and equilibrium pressures.

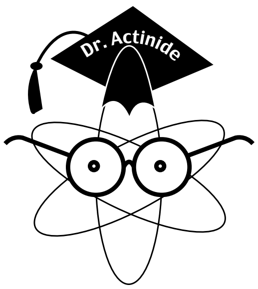
Research from the Shelf-Life Surveillance Project will be used by the DOE to ensure that representative stabilized materials in approved containers are safe in long-term storage.

The data compiled at Los Alamos will provide guidance on what may or may not cause a container to pressurize, help researchers understand the changes in gas composition over time, and explain the effect of corrosive gas generation in the cans.

The study also will provide data that will be used to establish a DOE sitewide surveillance program for the extensive amount of material packaged according to the DOE standard.■

Editorial

At the onset of the new millennium, Actinide Research Quarterly (ARQ) interviews Dr. Actinide for his views on events and trends unfolding in the coming years.



The opinions in this editorial are the author's. They do not necessarily represent the opinions of Los Alamos National Laboratory, the University of California, the Department of Energy, or the U.S. government.

Dr. Actinide's Plutonium Perspective

ARQ: *Since the beginning of the nuclear era in the 20^{th} century, you have enjoyed a monopolistic influence and authority over everything nuclear, based on your vast knowledge of actinides.*

Dr. Actinide: It has been said that a lot of intangible things ride on an individual's name: fame and fortune, status, character, and pedigree. The same things can be said about a hundred or so known elements in the universe. Some of these elements are born privileged. The actinide class of elements for which I speak is one of the privileged whose influence on the human race will not soon diminish.

ARQ: *Will plutonium go the way of Pluto in terms of its reputation?*

Dr. Actinide: A good question. As you know, man-made plutonium was born in the middle of the last century and named after the farthest planet in the solar system. Pluto has now fallen out of its planetary status and joined the rank of icy comets. For the past 50 years since its birth, man-made plutonium has enjoyed unmitigated attention from you in the eyes of its jealous elemental brethren. Will plutonium suffer the same degree of diminished status as its name source? Some spectacular comets visit Earth once in a lifetime, and then fade away for another generation to marvel at their sightings. So some may think of plutonium, with its cometlike visibility brightest during the past few decades. Whatever its reputation, plutonium will be around in the future, just as it has been around from the beginning of time.

ARQ: *What do you consider to be the major events of the past century in the area of actinide science?*

Dr. Actinide: It has been a little more than 50 years since the dawn of the nuclear age—a relatively short time. Clearly, one of the most significant developments in the 20^{th} century was the human race's recognition of the potential power of actinides, although there are other elements also worthy of recognition, like oxygen, which you need to live, and hydrogen, which fuels the sun.

ARQ: *It's just our habit of looking at things and events from a human perspective. Nonetheless, you will agree that the past 50 years have been eventful. Many events have occurred because of various applications of actinides.*

Dr. Actinide: Unquestionably so. The bomb-building race between the two world superpowers using uranium and plutonium captured people's imagination and threatened to destroy civilization as you know it. This has afforded me a certain degree of notoriety, but I am glad to see that some common sense prevails among the world powers today. They managed to reduce the nuclear threat during the last decade of the 20^{th} century. It also is commendable that during the last 50 years you have been able to extract some benefits from the peaceful use of actinides, namely as a source of nuclear power for the world's unquenchable thirst for energy. The choice between the two principal uses—nuclear explosive and nuclear energy—is entirely up to you.

ARQ: *Do you foresee any proliferation danger of nuclear weapons in this century?*

Dr. Actinide: Tools in human hands can be used either defensively or offensively. Tools in the hands of a madman will always present a threat. As long as individual nations consider that there is something to be gained by possessing nuclear weapons in terms of power, status, self-protection, superiority, or whatever, there will always be a nuclear proliferation threat. So far, there have not been any strong disincentives for possessing nuclear weapons, only attractiveness to some governments for their awesome destructive power to cover up other weaknesses. The world community will have to come up with a solution to this problem.

ARQ: *Do you have any thoughts on worldwide plutonium proliferation?*

Dr. Actinide: It's a well-known fact that what started out as a minute quantity in a test tube in one country about half a century ago has now grown to a thousand or more tons spread around some 30 countries. That's proliferation. The quantity of plutonium produced in nuclear reactors is increasing, and the number of countries that will produce more plutonium will also increase. You might say plutonium proliferation has continued unabated for the past 50 years.

ARQ: *We call that legitimate proliferation. How about other proliferation dangers, such as theft or production of nuclear materials by rogue nations?*

Dr. Actinide: You must keep in mind two things: quantity and frequency. Because more nations are producing more plutonium and processing it more frequently, it is very likely that at some point you may not be able to say that you have any control over the matter. That's the real danger.

ARQ: *You paint a fairly gloomy picture. Is the increased processing of plutonium all we should be worried about concerning plutonium proliferation?*

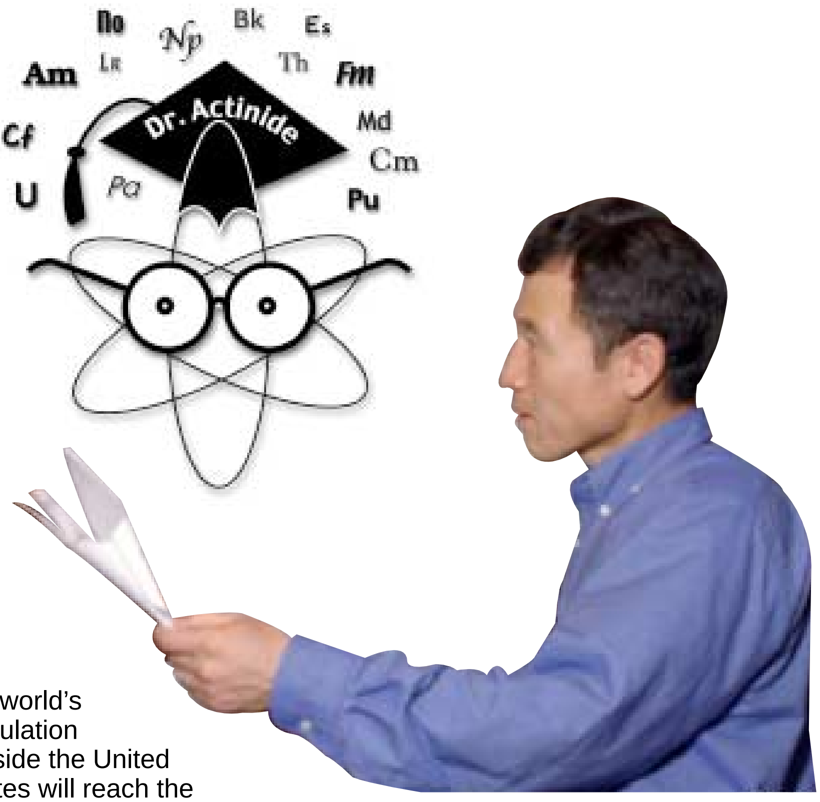
Dr. Actinide: Not entirely. Plutonium is a naturally occurring element. However, atmospheric testing of nuclear weapons has put a significant amount of plutonium into the environment. Continued processing of nuclear materials and accidental releases also deposit minute, but detectable, amounts of plutonium into the environment. This amount accumulates over many years because the major isotopic components of plutonium do not decay perceptively on the human time scale, except for the specific isotope plutonium-238, which is used in your space missions. In some cases, as in Russia, nuclear reactors in decaying abandoned submarines pose enormous potential bioenvironmental hazards.

ARQ: *Any thoughts on this new century?*

Dr. Actinide: In the first half of the 21st century, you'll face the daunting task of cleaning up the mess you produced during the last half-century. In addition, you'll have to safely manage the world's nuclear weapons stockpiles, while at the same time reducing the number of weapons. After accomplishing the cleanup task, and with some fresh air, the human race may develop a whole new perspective about itself and its surroundings. This new perspective may enable people to see that there are other purposes of human intelligence besides threatening each other with tools of mass destruction. But I'm afraid that you may not have the luxury of achieving this enlightened state before you have to deal with the more serious issue of a worldwide glut of plutonium.

ARQ: *A glut? Will it really be that bad?*

Dr. Actinide: As nations demand and consume more energy, they may not have much choice but to tap further into the vast potential energy source of actinides. Let's assume that in the distant future, 96 percent of



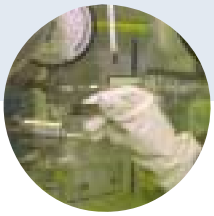
the world's population outside the United States will reach the same level of energy consumption as the United States, which currently consumes about 30 percent of the world's total energy. That's a 10-fold increase in world energy consumption. Regardless of whether this energy demand is met by fossil energy or nuclear energy, this scenario spells certain disaster. Add to that the projected world population growth, and the momentum for accelerated production of plutonium only increases. With the increased demand for energy by the growing world population, you will see a shift—plutonium will be less a proliferation concern and more a means to international cooperation toward better materials management. Better management of existing resources through conservation, efficient usage, and new development is the key to future prosperity for all. You also may be able to learn enough to harness nuclear energy from other elements like hydrogen, which is more abundant and perhaps less polluting.

ARQ: *Any final thoughts?*

Dr. Actinide: Many of the problems that you face or will face are common throughout the world. No nation alone has all the necessary resources and scientific knowledge to solve the world's nuclear problems, or to develop new energy sources. Nations will have to come together to help each other. It is a global problem.

ARQ: *How soon will we see this happening?*

Dr. Actinide: There is no telling. Your guess is as good as mine, but I can tell you this: You do not have to do anything to see the sun rise tomorrow morning, but if you do not plant the seed of a better tomorrow, you surely will not see a brighter future for following generations.■



Actinide Boride Ceramics Offer Alternatives for Safer Plutonium Storage

Los Alamos Team Investigates Low-Temperature Preparation Technique

Contributors to this article are: **Anthony Lupinetti** and **Eduardo Garcia** (NMT-2); and **Julie Fife** and **Kent Abney** (C-INC).

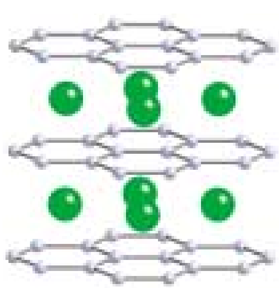
Safe storage of excess plutonium is the goal of a Los Alamos effort aimed at defining alternative actinide stabilization technologies. Under a treaty with the Russian Republic, the United States has declared approximately 38 metric tons of plutonium as excess to the nuclear weapons program; the Russians have declared a nearly equal quantity of material to be excess.

Photo by James Rickman

Anthony Lupinetti, left, of Actinide Processing Chemistry (NMT-2), and Kent Abney of Isotope and Nuclear Chemistry (C-INC) examine plutonium boride encased in a glass tube. The researchers are investigating ways to convert actinide metals into less-reactive, safer storage forms. Initial results indicate that actinide borides are stable in water, which should give them an advantage over plutonium metal or plutonium oxide as a storage form.

The majority of the U.S. material exists as highly purified metal, which poses a proliferation risk. It also poses a chemical risk because plutonium metal slowly reacts with water to form hydrogen if it is not properly stored. Over time this can result in overpressurization of the storage container or a buildup of flammable concentrations of hydrogen.

To overcome these and other issues, Los Alamos scientists are looking at new ways to convert actinide metals into less-reactive, safer storage forms. It has long been known that plutonium can be combined with boron, a solid semi-metal that shares some of the properties of metals as well as nonmetals, to form stable, water-insoluble plutonium borides. But these compounds could only be formed under high temperatures—in excess of 1,500 degrees Celsius. The Los Alamos researchers have explored a low-temperature method to create stable actinide ceramics. Initial results indicate that actinide borides are readily prepared and stable in water, which should give them an advantage over plutonium metal or plutonium oxide as a storage form.



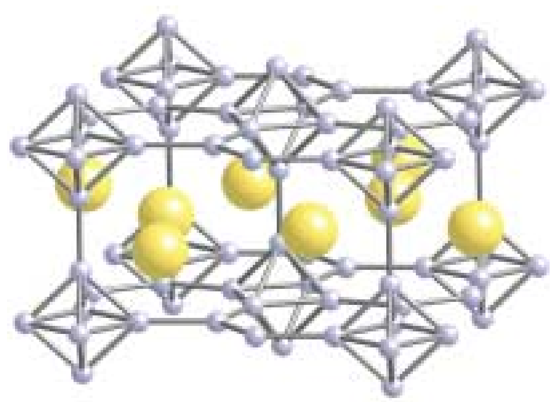
Plutonium and boron form seven different binary compounds. One of the compounds, plutonium diboride, is composed of plutonium atoms (green) sandwiched between two-dimensional networks of boron atoms (blue). This form of plutonium boride encapsulates a large amount of plutonium in a compact form (92 percent plutonium by weight). The team of Los Alamos researchers has succeeded in synthesizing this compound at low temperatures.

Actinide borides are typically composed of two- or three-dimensional arrays of linked boron clusters in which the metal atoms may be located between layers or encapsulated in the boron matrix. These networks create a class of materials with high plutonium content, high densities, and high melting points.

There are only 11 known binary plutonium, uranium, and thorium boride phases; examples include plutonium diboride, uranium tetraboride, and thorium hexaboride. These materials are known to be ceramics, but other chemical properties, such as reactivity, must still be explored.

In contrast, many transitional metal and lanthanide borides have been extensively studied. These borides have been used as nonreactive corrosion-resistant coatings. The Los Alamos researchers expect that many plutonium boride phases will also prove to be nonreactive, making them strong candidates as plutonium storage forms.

Until now, plutonium borides were made exclusively by high-temperature methods. To get the two elements to mix, plutonium metal



The structure of uranium tetraboride consists of uranium atoms (yellow) encapsulated in a three-dimensional network of boron atoms (blue). This ridged structure is one of the features that makes this compound such a stable storage form for actinide elements.

would be combined with varying amounts of boron, heated to greater than 1,500 degrees Celsius, then ground and reheated repeatedly until a homogeneous mixture was obtained. Another method to react plutonium with boron is arc melting, but this method produces even more extreme temperatures—above 3,000 degrees Celsius.

The new Los Alamos method uses a reactive process to combine actinide halides with reactive boride compounds that requires far lower temperatures—between 400 and 850 degrees Celsius—and doesn't require repeating the process over and over again.

The researchers began their experiments on a small scale by combining 250 milligrams of uranium tetrachloride with two equivalents of magnesium boride in a vacuum-sealed quartz tube and heating it to 850 degrees Celsius for one day. After the reaction, the tube is opened and the contents washed with water to remove the magnesium chloride byproduct and any unreacted uranium halide.

The result is pure uranium tetraboride, a stable, insoluble compound that is not easily converted to a pure actinide form that can be used in weapons. The uranium tetraboride was identified using powder x-ray diffraction.

Experiments were also performed to determine the effect of time, temperature, and excess magnesium boride on the reaction products. The major difference in these reactions is the crystallinity of the uranium tetraboride; longer reaction times and higher temperatures yield a more crystalline product. Higher crystallinity aids in the chemical characterization of the products by helping to identify other potential products.

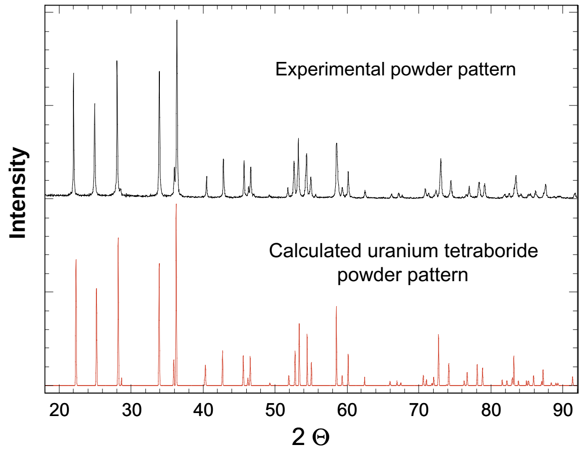
In addition to the studies using uranium tetrachloride, similar studies were carried out with thorium tetrachloride and plutonium trichloride, producing thorium hexaboride and plutonium di- and tetraboride, respectively. The researchers are also investigating ways to produce actinide borides at even lower temperatures by using fluxing agents like lithium chloride and potassium chloride, which when combined melt near 350 degrees Celsius.

The Los Alamos team is pursuing next-generation reactions that will allow them to further understand actinide chemistry and provide routes to other actinide ceramics including ternary materials containing plutonium, boron, and other elements like oxygen. Little is known about this class of actinide-containing three-component materials, and they are being investigated to broaden the fundamental knowledge of actinide materials science.

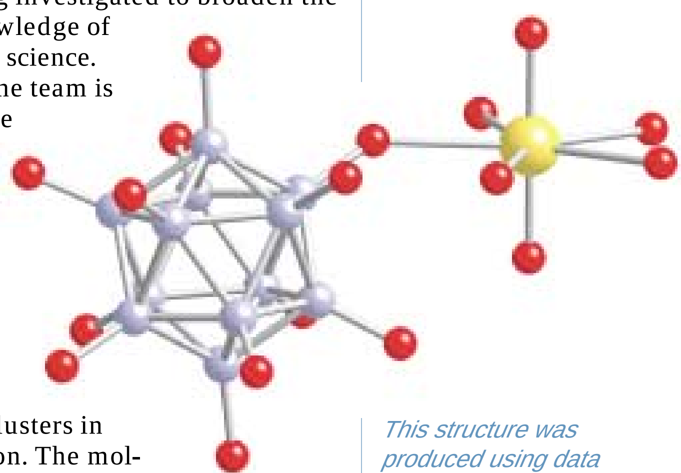
One avenue the team is investigating is the synthesis of molecular actinide compounds that contain novel boron-containing ligands, where the actinide is bonded to a boron cluster or clusters in a molecular fashion. The molecule is then heated to decompose the molecule and form a two- or three-dimensional array.

This approach allows for lower reaction temperatures and better control of the final product. As an example, the researchers recently synthesized a new uranium borane complex, which should be an excellent candidate for preparing new ternary actinide materials

The Los Alamos team presented their findings in April at the 221st American Chemical Society national meeting in San Diego.■

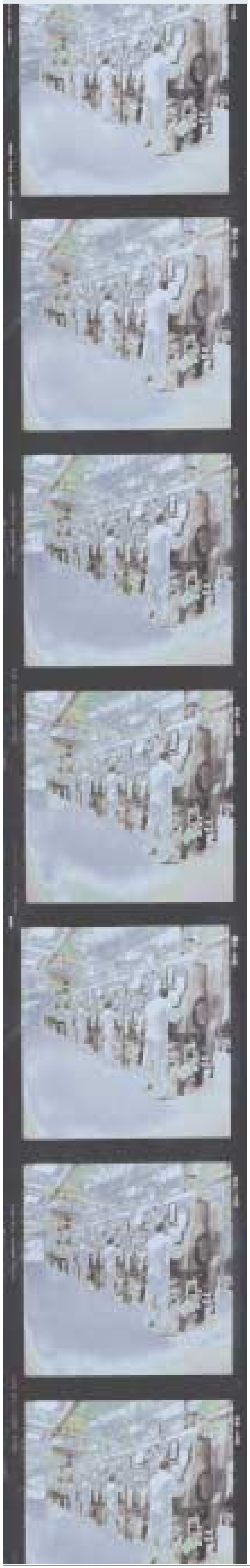


This graph shows two powder x-ray diffraction patterns. The top pattern is from the washed product of the reaction of uranium tetrachloride and magnesium diboride; on the bottom is a calculated pattern of uranium tetraboride. The two sets of data match exactly, confirming that uranium tetraboride is the only product.



This structure was produced using data from a single-crystal x-ray structure experiment. Uranium is shown in yellow, oxygen in red, and boron in blue. The hydrogen atoms bound to the cluster oxygen atoms (one each) and to the water molecules (two each) have been omitted for clarity. The Los Alamos team expects this uranyl borane compound to be a useful precursor to next-generation actinide boride storage forms.

NMT Division Review



Annual Review Rates NMT Division as "Outstanding/Excellent"

Division Commended for the Quality of Science and Its Vital Role in National Security

Nuclear Materials and Technology (NMT) Division has earned an outstanding / excellent rating in this year's annual division review. The final report commends the division for the quality of its science and notes the importance of maintaining and strengthening that science to support NMT's role in national security.

The division review, held in early May, was the first in two years; last year's was canceled because of the Cerro Grande Fire. The review covered the period July 1, 1998, through June 30, 2000. This year's review concentrated on weapons surveillance and stewardship, enhanced surveillance, components manufacturing, and dynamic testing.

"Many instances of outstanding performance were evident and the quality of the science and engineering had improved significantly compared to previous reviews," review committee members said in the report. "The overriding theme that emerged during the review is that future success in stockpile stewardship depends on the successful integration of research and manufacturing.

"It was evident that the division continues to perform work of vital importance to the U.S. nuclear defense program, especially with respect to the manufacture of pits," the report continued. "As the nation's only functional research and development facility for actinides that can handle pits, the division also supports a wide variety of other programs such as fuels and heat sources. Strong performance was noted in many areas."

The report cited NMT Division's wide range of activities, from fundamental research in plutonium to the actual fabrication of pits. "This integration of science and manufacturing, if well implemented, has the potential to be a model for the future activities of the Los Alamos National Laboratory … ."

The annual division review is a requirement of the University of California's contract with the Department of Energy. Under terms of the agreement, the Science and Technology Panel of the UC President's Council on the National Laboratories is responsible for assessing the quality of the science and technology programs at Los Alamos, Lawrence Berkeley, and Lawrence Livermore national laboratories.

At Los Alamos, director-appointed external committees, made up of experts in the various disciplines from the national laboratories, academia, and industry, review each technical division. The committees review about one-third or more of the divisions' programmatic activities each year, and all science and technology activities must be covered within a three-year cycle. Review committees base their evaluations on four criteria: quality of science, relevance to national needs and agency missions, performance in the technical development and operation of major research facilities, and programmatic performance and planning.

After the division reviews are completed at the end of May, the individual division reports are rolled up into a Laboratory-wide report called the Science and Technology Assessment, which is sent to the UC president's office at the end of August.

Review committee members and Los Alamos and NMT Division management attended an executive opening session May 8. Bill Press, Los Alamos deputy director for Science, Technology, and Programs, described for the review committee the Lab's vision, mission, and strategic business direction. He also discussed 10 top-level institutional goals for fiscal years 2001 and 2002, including the W88 pit production / certification goal, which applies directly to NMT Division's mission. The executive session gives Lab management the opportunity to provide some guidance to the review committee members as to what the Laboratory expects from them.

Carolyn Mangeng, deputy associate director for nuclear weapons, opened the first general session with a discussion of the Laboratory's weapons programs. She stressed the need to balance high-visibility programs, like pit production and certification, with the need to maintain the science base to attract new people and projects.

Mangeng mentioned several major successes in the stockpile stewardship program, including better characterizing of the aging of components in nuclear weapons; effectively using the Lab's Blue Mountain supercomputer for parallel processing; and obtaining new information from experiments on the dynamic properties of nuclear materials.

"Replicating Rocky Flats pits—a major component of NMT's stockpile stewardship program—is a challenge for the Laboratory and stockpile stewardship," said Mangeng. "There are many unknowns and it could prove to be a lot more difficult than first thought."

It's simply not possible to reproduce the manufacturing methods used at Rocky Flats, nor do we want to because of environmental and other considerations, said Mangeng. To come up with new methods that will result in an identical pit, to certify that the pits are identical, and to do this without nuclear testing will require technical know-how and research, support from across the Laboratory, and sustained funding from DOE.

NMT Division Leader Tim George followed Mangeng at the podium. "It's not a year I would have chosen to take over the division," he said, referring to several major contamination and security incidents that occurred in the division and at the Lab during 2000.

One incident, said George, proved to be a testament to the commitment of the people in NMT Division: their response to the Cerro Grande Fire. Division employees had NMT back up and running within two weeks of the Labwide closure and managed to maintain the time schedule set for pit production. In fact, the first pit produced following the fire was made on schedule, just weeks after the Plutonium Facility reopened, and a second pit was produced soon after.

George also discussed the important role Los Alamos has in the DOE complex because NMT Division maintains the nation's only remaining facility capable of handling all isotopes and chemical forms of plutonium, tritium, and other actinides. He also said that while a top priority of the division is the pilot production capability of pits, stockpile surveillance and enhanced surveillance may be the NMT mission most critical to national security.

Echoing Mangeng, George said that fulfilling the roles placed on NMT Division by the DOE and Washington will take additional support and funding.

The review committee's report commended George for his "high quality and accomplishments … . He has demonstrated his grasp of an exceptionally challenging job in a short period."

During the review, NMT Division's goals were laid out for the committee: stockpile management–pit manufacturing, stockpile stewardship–pit surveillance, environmental stewardship, nuclear materials disposition, energy and advanced fuels, and nuclear materials processing.

"Although these goals are realistic," said the report, "LANL and the DOE will not meet them without a major commitment of resources with support form the University of California. With the selection of LANL as the site for pit manufacture and surveillance for the U.S., it is extremely important that the NMT Division be provided with the facilities and resources to carry out the requisite actinide research, development, and manufacturing activities with compliance and safety."

The sessions continued through May 9 and included overviews on facility operations and the regulatory environment, NMT programs in general, the pit manufacturing project, weapons surveillance, emerging programs, components manufacturing, dynamic testing, the Seaborg Institute, other science highlights, and a report by the Science Leadership committee.

Technical presentation topics included last summer's international Plutonium Futures conference in Santa Fe, ion channel membranes for selective separation of actinide metal ions, plutonium electronic structure, and mechanical modeling for aged plutonium.

A video conference was held the afternoon of May 9 between the committee and top Lab management located in Los Alamos and DOE Defense Programs (DP) staff located in Washington, D.C. May 10 was devoted to private meetings between staff and the Division Review Committee, an executive outbrief session, and a closeout meeting open to NMT employees. The review wrapped up on May 11.

Anthony Rollett served as chairman of this year's review committee. Rollett is former deputy leader of Los Alamos's Materials Science and Technology (MST) Division. He currently is a member of the Department of Materials Science and Engineering at Carnegie Mellon University.

Other members of the review committee included Richard Bartsch, Department of Chemistry and Biochemistry, Texas Tech University; Rohinton Bhada, professor emeritus, retired, New Mexico State University; Darleane Hoffman, former leader of Los Alamos's Isotope and Nuclear Chemistry (INC) Division and currently a member of the Heavy Element Nuclear Radiochemistry Group, Lawrence Berkeley National Laboratory; and William Weston, Boeing Co., Rocketdyne Division. Susan Wood, vice president and director, Savannah River Technology Center, was unable to attend the review.

Participating as observers were former committee chair Ned Wogman, senior manager for national security, Pacific Northwest National Laboratory, and Lamar Miller, Department of Environmental Engineering Sciences, University of Florida.

NMT Division is in the process of recruiting several new members for the committee and hopes to have them on board in time for next year's division review.■

—Meredith S. Coonley

Anthony Rollett (far left), chairman of this year's Nuclear Materials and Technology (NMT) Division external review committee, shakes hands with K.C. Kim at the end of a day's briefings. Kim is NMT Division chief scientist and coordinator of this year's division review. To Rollett's left are Rohinton Bhada, committee member, and Lamar Miller, who served as an observer on the committee. Allen Hartford, director of the Lab's Science and Technology Base Programs (STB), is partially hidden behind Kim.

Photo by Mick Greenbank

Newsmakers

■ ***Mick Greenbank*** *of Nuclear Materials Science (NMT-16) recently won an award for his photograph "Down for the Count," shown at right. The photo placed second in the scientific-technical category at the 2001 Imaging Professionals of the Southwest Convention, held in Albuquerque in May. Greenbank's photo shows* ***Joe Riedel****, also of NMT-16, getting a whole-body survey in the In Vivo Measurements Laboratory, which provides direct measurements of radionuclides in Los Alamos workers. The 10-foot-by-10-foot chamber is located in a subbasement of the Health Research Laboratory (HRL). Riedel won a third-place award at the convention in the illustrative black-and-white category for an abstract view of pines and rocks titled "The Rocks Cry Out."*

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