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Fundamental and Applied Studies of Helium Ingrowth and Aging in Plutonium

Michael F. Stevens*, Thomas Zocco, Robert Albers, J. David Becker, Kevin Walter, Barbara Cort, Dennis Paisley and Michael Nastasi

Abstract

This is the final report of a two-year, Laboratory Directed Research and Development (LDRD) project at the Los Alamos National Laboratory (LANL). The purpose of this project was to develop new capabilities to assess the nucleation and growth of helium-associated defects in aged plutonium metal. This effort involved both fundamental and applied models to assist in predicting the transport and kinetics of helium in the metal lattice as well as *ab initio* calculations of the disposition of gallium in the fcc plutonium lattice and its resulting effects on phase stability. Experimentally this project aimed to establish experimental capabilities crucial to the prediction of helium effects in metals, such as transmission electron microscopy, thermal helium effusion, and the development of a laser-driven mini-flyer for understanding the role of helium and associated defects on shock response of plutonium surrogates.

Background and Research Objectives

The aging of both active and inactive weapons in the US nuclear arsenal is an acknowledged issue of great importance for the Stockpile Stewardship and Management Program of the DOE. Detailed knowledge of the mechanisms of aging and the consequences on component performance are important not only for predicting changes in overall physics package reliability, but are perhaps most important for proper scheduling of component changeout and sizing of the appropriate facilities for such refurbishments and replacement activities. In addition, detailed knowledge of stockpile materials, such as plutonium, is now needed to establish recertification and replacement standards. As new models for predicting the performance of nuclear weapons primaries are developed, experimental verification of the data used to establish the algorithms will be necessary. These experiments, consisting of dynamic and high-rate tests, or assessments of more fundamental characteristics of the materials such as interatomic potentials or elastic constants, will require a fundamental basis of understanding that links various length-scale structural features (both age-induced and intrinsic) to the physical and mechanical

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properties of interest. This project seeks to put in place many of the structure-to-property assessment capabilities that will be necessary to assess aging-induced changes to Pu microstructures.

Plutonium in a nuclear weapon ages primarily as a consequence of α-decay, resulting in an appreciable ingrowth of He and U in the microstructure. The other significant radiolytic ingrowth process involves the β-decay of residual ^{241}Pu to ^{241}Am. As of this point in time, little is known about the metallurgical disposition of these species in the lattice. These elements may directly alter the microstructure, by chemically stabilizing a new phase, or they may exacerbate the inherent phase instability of plutonium alloys. Alternatively, secondary effects due to point defect damage resulting from the radiolytic decay processes may give rise to structural alterations which may degrade the physical integrity of a component or alter its properties during detonation. Structural changes may manifest themselves as phase transitions, physical property changes, or changes in the dynamic response of the material during device performance.

Importance to LANL's Science and Technology Base and National R&D Needs

The work described herein and its continuation is motivated by a need to more fully understand the relationship between the physical and mechanical properties of plutonium and its underlying microstructure. As stated above, this is needed for knowing the levels on which plutonium parts must be examined in order to certify (for new) or recertify them for continued use. In the case of new materials, we must know how to compare the properties of newly fabricated plutonium with plutonium that was fabricated using well known schemes at the Rocky Flats Plant. For continued use of existing materials, which is the focus of this research, we must know how storage in the stockpile and radioactive, chemical and metallurgical events are altering the as-fabricated microstructure. The technical issues about which we construct this study are as follows:

- What is the disposition of radiolytically ingrown helium in the plutonium lattice? We must develop a better knowledge of the metallurgical behavior of helium in the host, and its interaction with other point defects, long-range diffusion and bubble precipitation tendencies. Such knowledge will allow us to ultimately predict whether helium bubbles, point defect void swelling, accumulation along grain boundaries, or some other distribution of helium is likely to cause physical or mechanical property changes.

- Will radiolytic decay-induced defects and chemically active species ingrowth cause phase instability in the plutonium matrix? This really addresses the question of phase stability in δ -phase plutonium. We must begin to understand how species such as Ga, Al, Si, and even Am stabilize this phase from an electronic bonding point of view, in order to predict how local alterations in structure and chemical composition may affect phase stability.
- How can we simulate the effects of radiolytic aging in plutonium in order to measure the effects of advanced aging on plutonium properties? Besides the obvious advantages of measuring plutonium alloys at advanced states of age, such techniques are very useful for creating surrogates that can be studied at various characterization facilities with much less preparation and caution than the actual plutonium samples, thus providing low-cost development of enhanced surveillance techniques.
- Can we develop a laboratory-scale facility for directly measuring the dynamic and shock properties of plutonium and other weapons materials? Up to now, we were required to use large-scale flyer plate and other techniques to study plutonium under shock conditions. New techniques, such as the laser-driven mini-flyer plate, offer the possibility of studying the dynamic response of plutonium in small quantities, if the technique can be developed to a sufficient level and the data corroborated by an independent technique. Ultimately, such studies are necessary to establish when microstructural changes alter properties that are of concern to the weapon physicist. Such properties include the elastic Hugoniot, melting on the Hugoniot, departures from the Hugoniot resulting from transformation, dynamic strength, and spall.

This project continues and strengthens a Laboratory competency in the materials science of the actinides (i.e., nuclear and advanced materials) for which it has been known as a world leader. This research will establish strong, interdisciplinary studies of the physics of defects in metals, stronger understanding of phase stability in materials, and advanced techniques for the evaluation of high stresses and shock waves in metals, in particular plutonium.

At the same time, this proposal focuses these materials science strengths on a highly visible problem in nuclear weapons science, namely, the aging of plutonium in stockpile weapons and resulting property changes. Success in this project (and related programmatic efforts) will ensure that weapons stewards are not surprised by aging events altering the integrity of pits. Also, this work will assist in the understanding of dynamic deformation

and shock behavior of plutonium, necessary for advanced simulations without nuclear testing.

Scientific Approach and Accomplishments

The technical work that comprised this project was conducted as separate, but coordinated studies of the radiolytic aging of plutonium. Each component will be described as a fully independent section.

A. Atomistic Modeling of Plutonium Aging (Albers)

The goal of this part of the project has been to microscopically model the effects of the radiolytic decay of Pu into U and He on aged Pu. We have focused on microscopic aspects of the aging processes.

A proper treatment of these aging mechanisms involves modeling the damage done by the recoiling U and He atoms during the radiolytic decay process, and the subsequent effects of the U and He ingrowth on the mechanical properties of aged Pu. Of particular interest is the swelling seen in related materials (e.g., radiation-damaged stainless steels) and the effects of possible helium embrittlement. Of additional interest (but not studied here) is possible phase transformations caused by such ingrowth.

To model these types of phenomena, it would be extremely useful to have much more experimental data than the meager amount that is currently available. In the absence of this data, we have tried to proceed with the best first-principles approach models available with today's technology, with the hope that we can later validate this approach as the amount of experimental data increases.

To have believable and quantitative models, the key aspect is the quality of the atomic potentials between the different types of atoms. For metallic systems, the best atomistic models are currently embedded-atom potentials (EAM). For this reason we began this project by attempting to develop new EAM potentials for Pu. Over the course of the project we have developed two new generations of EAM potentials for metallic Pu.

The chief obstacle in developing potentials for Pu is the very unusual electronic structure, which is dominated by its f-electron character. This has two significant consequences.

1. LDA calculations inapplicable to delta phase. The fcc phase of Pu (delta) is the phase of greatest interest because in its stabilized form (e.g., with small additions of Ga or Al) it has the best mechanical and metallurgical properties. The normal procedure for developing EAM potentials is to use the highest quality local-density-approximation (LDA) electronic-band structure calculations or experimental data to fit the parameters needed for

the phenomenological EAM potentials. For delta Pu this is very difficult because of the sparcity of available experimental data. What makes the situation even more serious is that LDA calculations cannot make up for this lack of data, since they are inapplicable to the delta phase. Due to strong intra-atomic electron-electron correlations, the f-shell in the actinides undergoes a metal-insulating -like transition whereby the f-shell localizes at Pu. For actinide elements lighter than Pu, the f-shell is fully itinerant and participates in the bonding properties of the material. For the next heavier element (Am) and beyond, the f-shell is localized like the rare-earth series and does not participate in the bonding. Pu is at the cross-over point and demonstrates this by having very complex bonding properties of the f-shell, for which there is no adequate first-principles theory.

For the high-density low-temperature phase (alpha), the f-electrons are normal bonding electrons and conventional LDA theory works very well. For the delta (and other phases) of Pu, the correlation effects manifest themselves by partially turning off the bonding properties. Based on extrapolations from Am and LDA theory where the localization of the f-electrons are simulated by removing their bonding properties, one can estimate that approximately 1/2 of the f-bonding strength is turned off in delta Pu. Since these types of correlations go well beyond the approximations made in LDA theories, the normal LDA band-structure approach is completely unreliable for the delta phase. For example, any estimate of the volume per atom of an LDA calculation of Pu in the fcc (delta) crystal structure yields the high density of the alpha phase instead of the greatly expanded volume of the delta phase. For this reason we have an incomplete data set to determine EAM type potentials. To get around this problem we have tried to scale the properties of other similar metals. This was done for the first EAM potentials where Ni was used as a reference material (in addition to measured delta-phase properties). In the second class of EAM potentials we changed reference materials to Al, since this fits the stress-strain curves much better, and forced the elastic constants to match the extreme anistropy measured in the only experiment done for delta Pu.

2. Strong directional bonding of potentials. The second problem with Pu potentials is their expected strong directional bonding. In metallic transition-metal materials, it is well known that EAM potentials have problems fitting a variety of anomalous phonon and structural properties because they are inherently isotropic and do not include directional bonding. With the very strange distorted crystal structures of Pu, directional bonding is expected to be even stronger. For this reason we are developing a new type of tight-binding approach for atomistic potentials under the Accelerated Strategic Computing Initiative (ASCI) program, which should be able to handle strong directional bonding. Our hope was to apply these new potentials to calculate the aging mechanisms once they were

validated and became available. In this LDRD project, we were initially trying to understand the aging mechanisms as well as possible with the less accurate EAM technology and building the calculational methodology and tools needed to study these types of phenomena, and then ultimately improving our calculations by plugging in the better tight-binding type of potentials. Unfortunately, we were unable to complete this switch-over during the two-year term of our project.

With the current Pu EAM potentials that we have developed, we have calculated phonon spectra in the delta phase in different crystallographic directions. We have also attempted to calculate clusters of vacancies in delta Pu in the belief that this would be a likely place for the helium to end up. Our calculations (Fig. 1) indeed show that clusters of a few vacancies are more energetically favorable than single vacancies or huge clusters of vacancies (which would simulate cracks in the material). These results are consistent with positron annihilation studies done at Lawrence Livermore National Laboratory (LLNL), which seem to indicate that the helium is distributed in either single vacancies or very small vacancy clusters.

Our next task was to attempt to calculate the effects of the helium in the lattice. Because there is no experimental data at all with which to fit the helium-Pu cross potentials (although there is very good data on pure He to fit the helium-helium interactions, which are also needed), we undertook a set of very computationally expensive LDA band-structure calculations. Although these calculations include the full f-bonding effects in the potential (since they are LDA calculations), our approach has been to proceed anyway, since no other choice is possible. Once the fully f-bonded potentials are finished being fitted, we will attempt to scale out some of the f-bonding strength of the potentials so that we can attempt to fit the delta phase of Pu.

To fit the cross terms we have done calculations on hypothetical PuHe in the CsCl structure and PuHe3 in the Cu3Au crystal structure. For each structure we calculated the cohesive energy, theoretical lattice constant, and bulk modulus (at the theoretically predicted minimum of total energy). We also did similar calculations on fcc and bcc Pu with the same basis function set so that we could look for differences caused by the helium without mixing different types of calculations. The bcc phase was also chosen since, if helium atoms in the CsCl structure are replaced with Pu atoms, the lattice reverts back to a bcc structure. The Cu3Au structure becomes fcc if all the helium atoms are replaced with Pu atoms.

We are currently in the process of fitting the fully f-bonded cross potentials. Once these potentials have been scaled down for the delta phase, we will then be able to add the helium to the small vacancy clusters examined earlier and to test the energetically

favorableness or lack thereof of different vacancy-helium configurations. We will then also be able to examine diffusion characteristics of helium (e.g., diffusion barriers in the fcc crystal structure), and a large number of other processes important for modeling the effects of radiolytic decay.

B. *Ab Initio* Calculations of Phase Stabilization in δ-Plutonium (Becker)

The strategy for these calculations was to perform fully quantum-mechanical calculations on Pu compounds relevant to the stability of the δ alloy with respect to the α and Pu_3X phases. The full-potential linearized muffin-tin orbital (LMTO) method in the local density approximation is used to calculate the self-consistent band structures and total energies of selected crystal structures. To model a dilute Pu-Ga alloy a 32-atom supercell, equivalent to 2×2×2 fcc unit cells with a Ga atom at the center, is chosen as the starting configuration. The supercell volume is fixed at the LDA theoretical atomic volume of δ Pu, namely 16.4 $Å^3$. Using the LMTO method, the forces on the nearest (Pu) atoms to Ga in a $Pu_{31}Ga$ supercell are calculated. Starting with a perfect fcc lattice, atomic positions are adjusted to minimize the forces using a linear projection technique.

The results are summarized as follows. The nearest-neighbor shell surrounding the Ga atom relaxes inward by approximately 1% of the starting bond length, 2.86 Å. The Pu atoms in the third shell relax away from the Ga atom by 0.1%, though constrained by symmetry to move in the planes of the supercell faces (see Figure 2). The Pu atoms in the 2nd neighbor shell around Ga are fixed since they are equidistant between pairs of Ga atoms. The nearest-neighbor shell around these atoms relaxes anisotropically with 4 of their nearest neighbors moving outward (and toward the Ga) by 1%. EXAFS data for Pu-3.3 at% Ga imply an inward relaxation of the Pu shell surrounding a Ga atom of 3.5%.

While the LDA accurately predicts the ground state volume of the monoclinic α phase of Pu, no treatment of the δ phase has predicted the large increase in volume between the α and δ phases (19 $Å^3 \Rightarrow$ 25 $Å^3$). Thermal expansion is not responsible for the volume increase, and the volume of the supposed cubic ground state structure of Pu_3Ga is commensurately underestimated by *ab initio* methods. This shortcoming probably lies in the treatment of the Pu *f* states. In the lattice relaxation calculation the *f* states are allowed itinerant behavior. When the *f* states are constrained to fully localized behavior, the calculated δ–Pu volume jumps to 29 $Å^3$. We have explored the use of an augmented

Hamiltonian in which a certain number of f states are confined to localized energy levels while allowing some f participation in the bonding. The results of this treatment are encouraging. The proper volume for δ Pu is predicted, and the application of the method to several rare earth elements has also yielded accurate volume estimates.

At room temperature or higher, Ga-rich (i.e., concentrations of Ga above 13%) δ–phase alloys are in equilibrium with a Pu_3Ga compound which has the $L1_2$ (Cu_3Au) structure. An understanding of the thermodynamic behavior of the Pu_3Ga phase is therefore critical to understanding the stability of the δ phase. The alloying effects of the other Group IIIB metals with Pu are very similar to those of Ga. Each forms a stabilized δ phase in equilibrium with an $L1_2$ compound at Pu_3X. We have studied these four compounds: Pu_3Al, Pu_3Ga, Pu_3In, and Pu_3Tl, systematically with the full-potential linearized muffin-tin orbital (LMTO) method. The results of this study show that the behavior of the four alloy systems fall into two regimes of electronic behavior. Pu_3Al and Pu_3Ga have nearly identical average atomic volumes (both computed and measured) that is less than the atomic volume of the pure Pu δ phase, and the calculated formation energies of these compounds are much more negative than the other two compounds, Pu_3In and Pu_3Tl. The latter compounds have nearly identical atomic volumes greater than that of δ Pu. The two behavior regimes seem to "straddle" the behavior of pure δ Pu. An important clue to this dichotomy is found in the electronic densities of states of the compounds and of Pu. Pronounced hybridization in the second regime compounds (Pu_3In, and Pu_3Tl) possibly signals the incipient phase instability of the compounds and, thus, the less stable formation energies and larger atomic volumes typical of the onset of delocalized f electron behavior and open, low-symmetry crystal structure.

C. He Effects on Dynamic Properties of Pu and Its Surrogates (Walter)

The primary goal of this task was to find a surrogate material for δ-Pu that could be used in the study of the effects of radiation damage on mechanical properties. The surrogate would then be irriadiated, using an ion beam, to study the effects of the 5.4-MeV α and the 85-keV U that result from spontaneous decay of ^{249}Pu. Extensive computer simulations using the Transport of Ions in Matter (TRIM) code were used to specify ion irradiation processes to mimic the radiation damage in the surrogate and make it as close as possible to that thought to occur in self-irradiated Pu. Mechanical properties of the

surrogate, both before and after irradiation, would be measured by nano-indentation techniques that give hardness and elastic modulus information. As preliminary results were compiled, the goal of the work shifted to making irradiated samples of surrogate material for dynamic-properties testing using the laser-driven mini-flyer. As of this writing, several samples have been made and testing is imminent.

Surrogate material development

Two important material properties for studying radiation damage of metals are crystal structure and mass-density. Different populations and types of radiation-induced defects can occur for different crystal systems. It is also important to study a homogenous material that does not contain secondary phases with different crystal structure or composition. Second, a material's mass-density affects the distance an energetic particle travels through a solid, and thus affects the concentration of radiation defects. δ-Pu has a mass-density of 15.8 g/cc and a face-centered cubic structure. The Au-Ag system was chosen as a suitable surrogate because of its complete miscibility and its fcc crystal structure. Secondly, an alloy of 60 at% Au and 40 at% Ag has a mass density of 15.8 g/cc. Two methods, electron beam evaporation and vacuum arc-melting, were used to fabricate the surrogate material.

Electron beam evaporation onto Si was one of the methods used to fabricate thin films of Au/Ag. However, the coatings were generally thin (< 1 μm) and the coating composition and density varied from the desired values. Irradiation of the coatings resulted in significant sputtering. Nano-indentation tests on the irradiated samples did not indicate any change in the hardness or modulus of the coating. After dynamic-properties testing was deemed important, which requires free-standing material at least 25 microns in thickness, electron evaporation of the surrogate was abandoned.

A plug of 60 at% Au and 40 at% Ag was made by vacuum arc-melting. The composition, as measured by ion-beam analysis, is 57 at% Au and 43 at% Ag. The plug was cold-rolled to 75 microns in thickness and then annealed in air at 800°C for 120 minutes. The resulting grain structure is shown in Fig. 3. After annealing, samples with the correct dimensions were fabricated for the laser-driven miniflyer tests.

Irradiation Simulation and Experiments

The TRIM code was used to simulate the irradiation of Pu and the Au/Ag surrogate. One of the key input parameters needed for TRIM is the displacement energy, E_d, which is the energy that must be transferred to a Pu atom to displace it from its lattice site and create a vacancy. The number of displaced atoms, the number of times an atom is displaced, and the vacancy concentration are all dependent on E_d. A significant effort was made to find

published values for E_d of Pu in both the unclassified and classified literature. Only one estimate was found; however, the method used to estimate E_d for Pu is dubious. We used a more established method of estimating E_d by using the known correlation of the enthalpy of sublimation and E_d. Figure 4 shows a graph of fcc metals, their heat of sublimation and their measured displacement energies. The correlation is obvious and was first noted by Terry Mitchell back in the 1970s. The heat of sublimation for Pu is 3.6 eV/atom , which indicates an E_d of 33 eV.

The estimated value of E_d was used in the TRIM calculations to estimate the number of displacements that occur during spontaneous decay of ^{239}Pu. The simulations indicate that

- a 5.63-MeV α into Au/Ag gives the same displacement distribution as a 5.1-MeV α in Pu and it takes 30% less alpha particles to get the same number of displacements in Au/Ag as occurs in Pu (see Fig. 5), and
- a 80-keV Xe ion in Au/Ag gives the same displacement distribution as 85.8-keV U into Pu, and it takes 5% less Xe ions to get the same number of displacements in Au/Ag as occurs in Pu due to U irradiation (see Fig. 6).

The simulations show that it is possible to engineer surrogate materials to have the same damage distribution as self-irradiated ^{239}Pu. However, as the research direction shifted to dynamic testing of materials, such irradiated surfaces were not of primary interest and thus were not produced. If the need for such samples arises in the future, we already know how to do produce such samples.

Laser-Driven Mini-Flyer (LDMF) Experiments

The LDMF experiments were designed to test the spallation strength of the Au/Ag surrogate before and after He irradiation. The simplest experiment was to design the samples so that the tensile stress reached a maximum at the center of the sample, and that the maximum He concentration also occur at the center of the sample. The highest He ion energy available is 9 MeV, which has a range of about 20 μm. Thus, the Au/Ag sample thickness should be 40-45 μm. Specifics of the LDMF experiment require the flyer plate to also be the same materials as the sample (Au/Ag) and have one-half the thickness, or 20 μm. Au/Ag samples for irradiation and spallation tests were mechanically polished from the 75 μm tapes to a 40-μm thickness. Polishing was done using standard TEM sample preparation techniques. Flyer plates, 3-mm diameter, were punched from Au/Ag tapes cold-rolled to 20-μm thickness. Irradiated samples were made with a He concentration equivalent to 20 and 40 years of spontaneous ^{239}Pu decay. These and other unirradiated samples are awaiting testing.

A TRIM simulation of the expected He concentration profile in the Au/Ag samples is shown in Fig. 7. The concentration shown represents about 20 years of Pu self-irradiation. The same He concentration can be achieved using the Tandem accelerator in the Los Alamos Ion Beam Materials Laboratory (IBML) in about 12 hours over a sample area of 1.5 cm^2.

The important materials parameters for modeling the response of a material to LDMF testing are the mass-density, thickness, elastic moduli (shear and longitudinal), and the yield stress. The mass-density of the Au/Ag alloy was designed to be 15.8 g/cc. The sample thicknesses were designed according to our irradiation capability as discussed above. The elastic modulus was measured using nano-indentation techniques. The results, shown in Fig. 8, give an elastic modulus of 80 GPa. The hardness results are shown in Fig. 9. As shown, the hardness is ~0.9 GPa. For most metals, the yield stress is one-third the hardness. Thus, the yield stress for the Au/Ag surrogate materials is 300 MPa.

If the He irradiation resulted in He bubble formation, the behavior under LDMF testing could vary because of the locally reduced mass density and induced strain. A cross-sectional TEM specimen was made of the irradiated Au/Ag material irradiated at the higher dose in order to simulate 40 years of Pu self-irradiation. A few "structures" that could be bubbles were seen, but they were not uniformily distributed. It is not certain if the bubbles resulted from He irradiation or Ar-ion beam milling during the sample preparation. The bubbles were several hundred nanometers in diameter. Due to problems with the microscope, no pictures or diffraction patterns were taken. At this time, there is no conclusive evidence to suggest that the He irradiation resulted in He bubble formation in the Au/Ag surrogate.

Publications

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Becker, J.D.; Cooper, B.R.; Wills, J.M.; and Cox, L., "Calculated Structural Disorder in δ-stabilized Pu," to appear in *J. of Alloys and Compounds* (1997).

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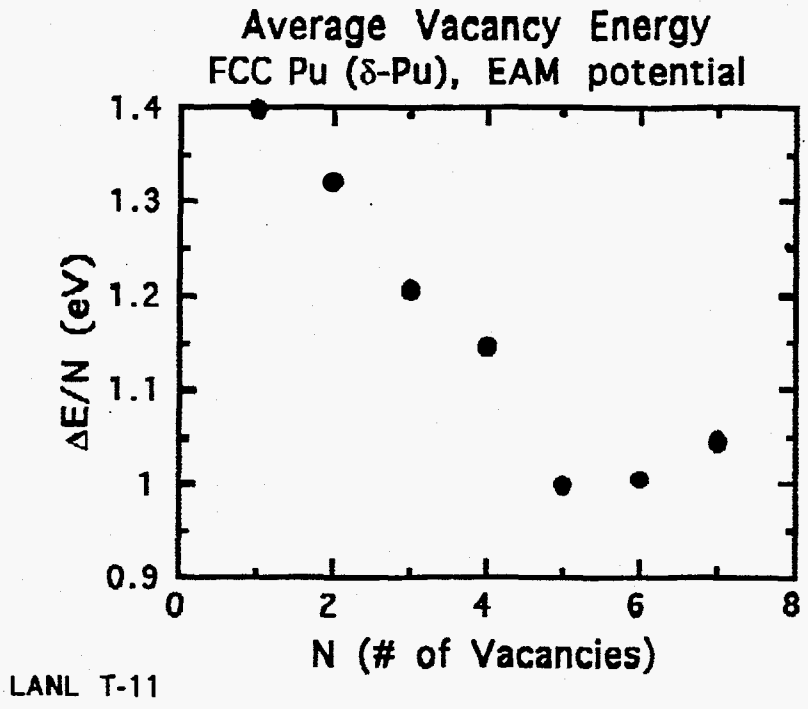

Figure 1. Average calculated vacancy cluster energy as a function of size.

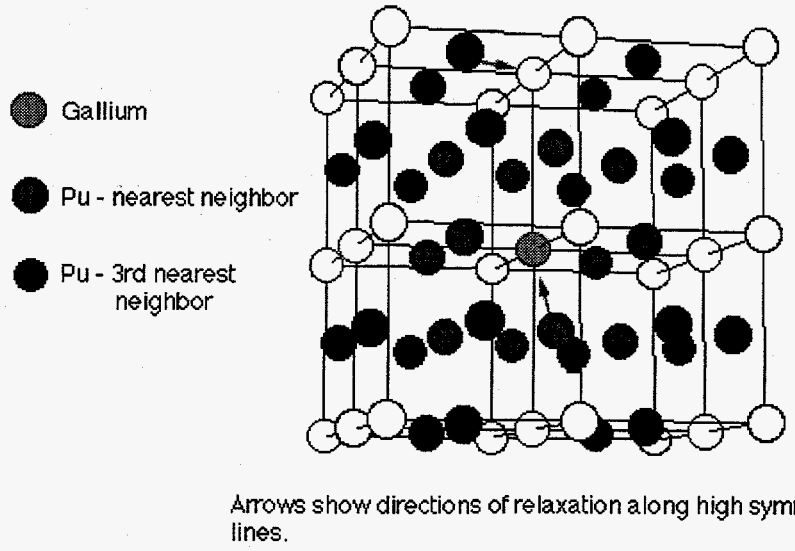

Figure 2. 32-atom supercell used to examine relaxations around Ga atoms in the Pu matrix.

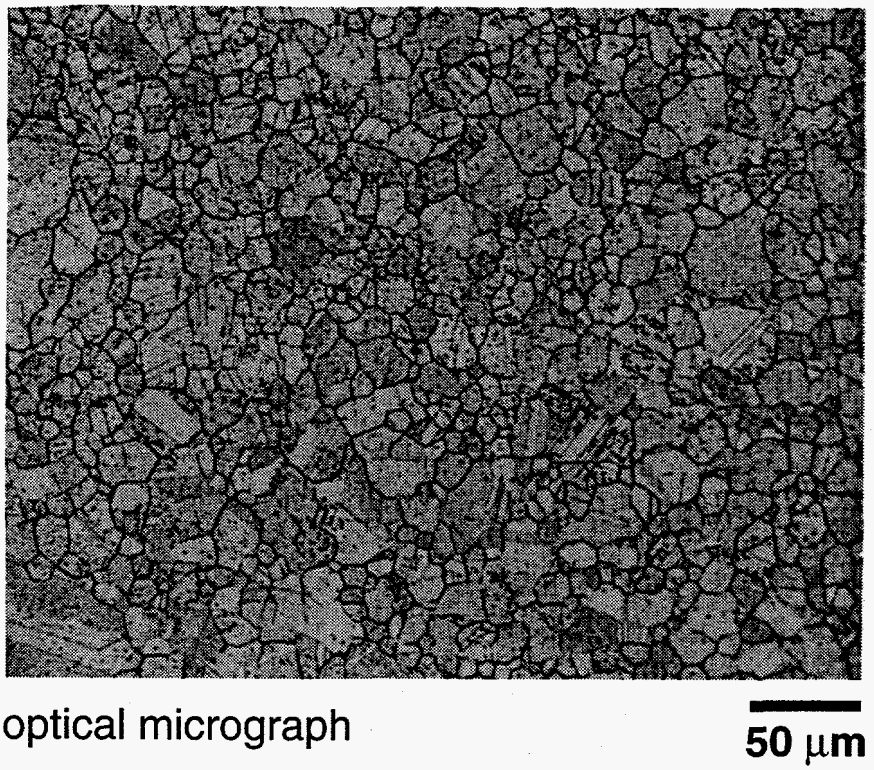

Figure 3. Optical micrograph of the Au/Ag surrogate material with a grain diameter of ~18 μm, and showing a few coarser grains and annealing twins.

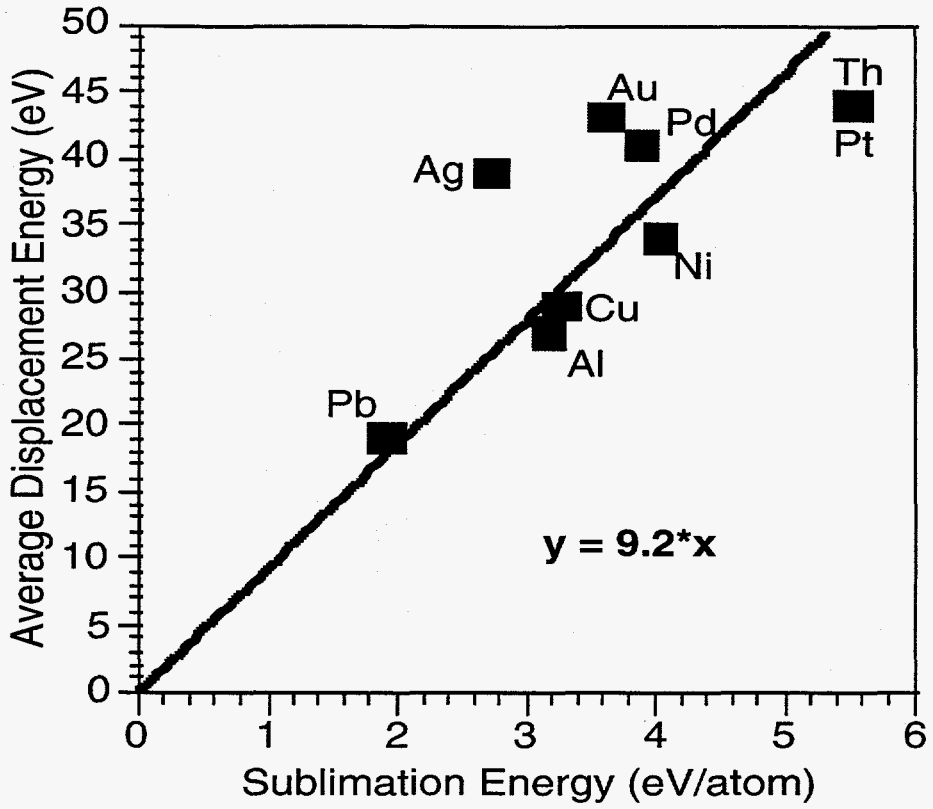

Figure 4. Correlation of the energy of displacement and the heat of sublimation for fcc metals.

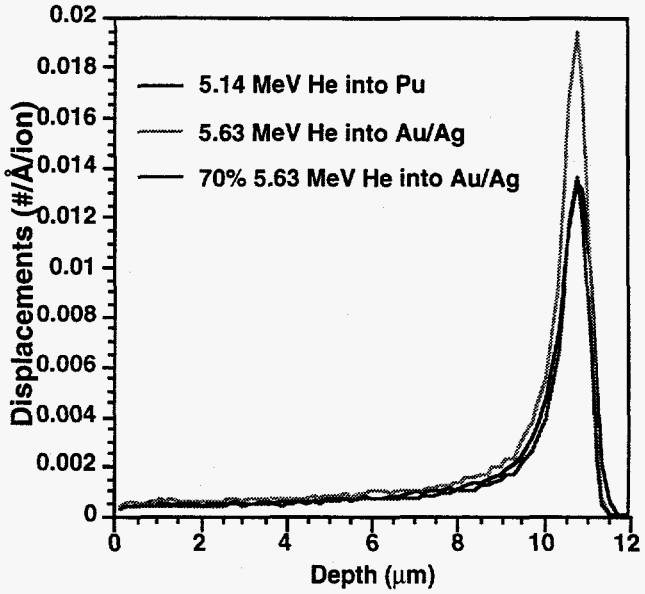

Figure 5. TRIM simulation of He irradiation of Pu and the Au/Ag surrogate. The results show that it takes a higher-energy α particle, and 30% less αs, to get the exact same displacement distribution.

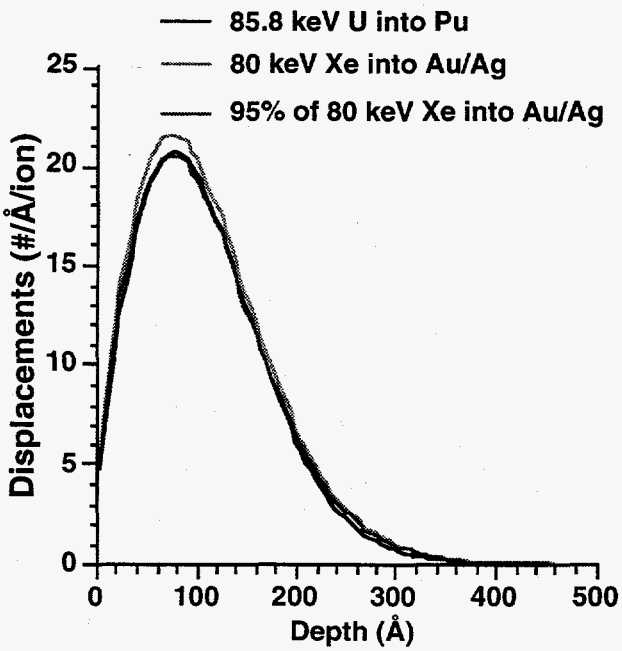

Figure 6. TRIM simulation of U irradiation of Pu and the Xe irradiation of the Au/Ag surrogate. The results show that it takes a lower energy Xe ion and 5% less ions to get the exact same displacement distribution.

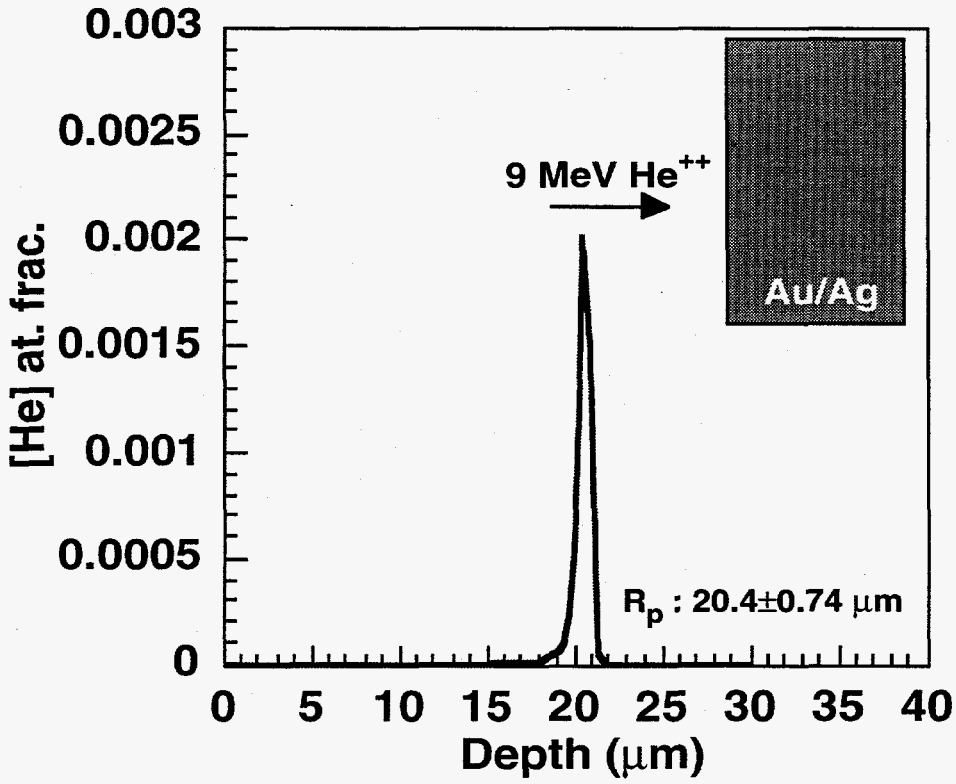

Figure 7. TRIM simulation of the He concentration profile for the irradiated Au/Ag samples.

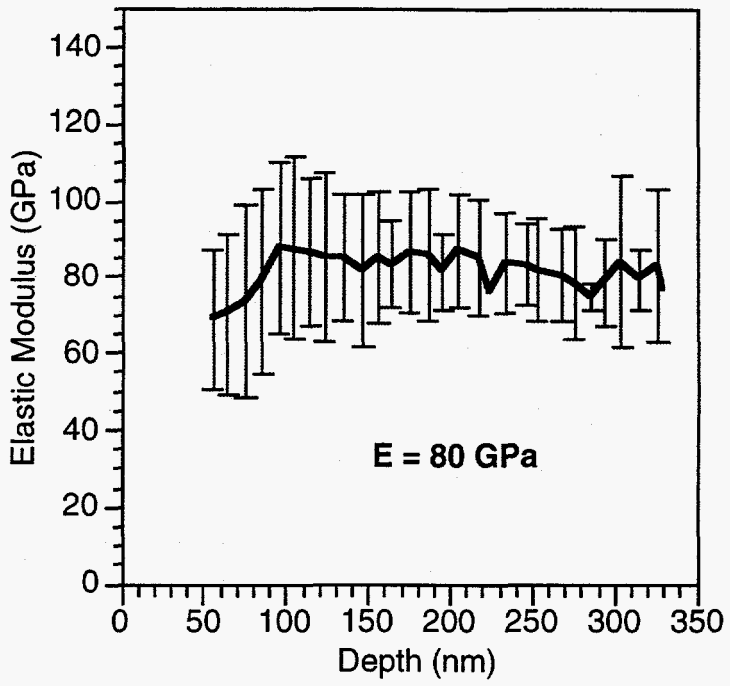

Figure 8. Elastic modulus measurement of 60 at% Au—40 at% Ag alloy as measured by nano-indentation.

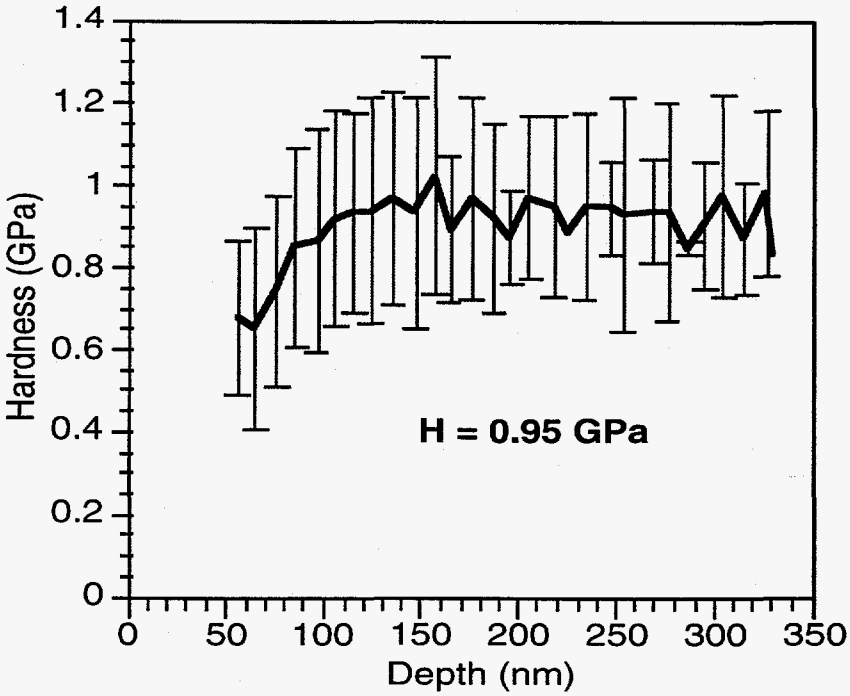

Figure 9. Hardness measurement of 60 at% Au—40 at% Ag alloy as measured by nano-indentation.