UCRL--52000-90-10

DE91 004680

■ October 1990

Scientific Editor

Kent C. Johnson

Editorial Staff

Lauren de Vore
Robert D. Kirvel
Kevin Gleason
Nona M. Sanford

Art Staff

Lynn M. Costa
Kathryn House
Janet Crampton-Pipes
George Kitrinos
Paul M. Harding

Compositor

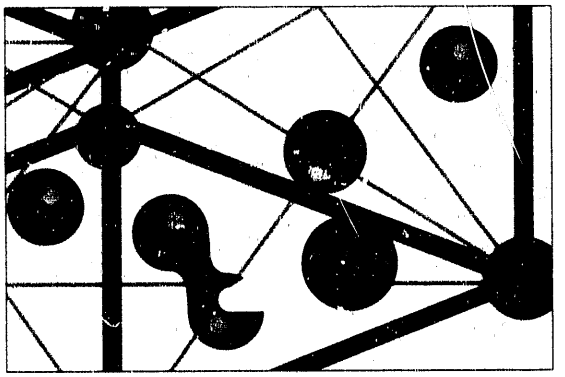
Louisa Cardoza

Printed in the United States of America
Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22161
Price codes: printed copy A03, microfiche A01

UCRL-52000-90-10
Distribution Category UC-700
October 1990

Energy & Technology Review

History of Cold Fusion Experiments



Sparked by the announcement March 24, 1989, of a new nuclear fusion process, scientists around the world quickly attempted to reproduce the results. Few other efforts in recent scientific history have rivaled the level of activity created by the cold fusion controversy.

For more than thirty years, research aimed at making controlled thermonuclear fusion reactions in the laboratory has been carried out with considerable success throughout the world. In the past, consistent government funding at fairly high levels has been provided in response to the promise of energy from the inexhaustible fuels of the fusion process. The pursuit of energy from fusion reactors has enjoyed favorable public response because this energy source is viewed as being safe and environmentally acceptable.

On the other hand, the technical challenges associated with developing fusion energy as a practical power source are considerable, and a substantial effort into the next century will be required. The recent Fusion Power Advisory Committee to the DOE strongly supports this effort, despite high costs and uncertainties, since the promise of fusion is breathtaking.

The startling announcement of a new nuclear fusion process made possible by an inexpensive device using conventional materials, made on national television on March 24, 1989, by Dr. Martin Fleischmann and Dr. Stanley Pons reached a highly receptive audience. The implication of their claim was that unlimited, cheap energy could be available soon. As shown in Figure 1, their table-top electrolytic cells consisted simply of a pair of electrodes immersed in a beaker of heavy water (D_2O) containing lithium deuteroxide (LiOD) in solution and connected to a battery. Small power-generating units were indicated, and the device could be put together easily with conventional materials at low cost. The heat produced in their cells, according to the first newspaper reports, was 26 W/cm^3 from palladium rod, or about four times more power than was put into the setup. Moreover, this heat did not have the expected level of accompanying neutrons that would emit radioactivity into the surroundings. The press release triggered an immediate and expected response: scientists around the world rushed to their laboratories to attempt to reproduce the results.

Analyzing the Underlying Mechanisms

To fuse the isotopes of hydrogen and thereby release an amount of energy, as given by the famous Einstein relation, the atoms must

be brought very close together. The probability that hydrogen isotopes will fuse increases dramatically as the distance between atoms decreases. [Deuterium (D) nuclei are hydrogen nuclei with an atomic mass of 2, and tritium (T) nuclei are hydrogen nuclei with an atomic mass of 3.] In a room-temperature gas of deuterium molecules bound together by electrons, the spacing between atoms is 0.074 nm, and the fusion rate per pair of atoms is about 10^{-74} reactions per second, an extremely low value. This atomic spacing can be reduced about 200 times by replacing an electron with a muon. A muon also has a negative charge but is about 207 times heavier than an electron. In muon-catalyzed fusion at room temperature, fusion reactions can occur at rates up to 10^{10} reactions per pair per second.[1] Because investigators were faced with the problem of explaining reported cold fusion rates (based on neutron production inferred from gamma measurements) in the range of 10^{-18} per pair per second, there was immediate speculation that deuterium imbedded in palladium metal allowed the necessary close spacing.

In the conventional fusion approach, termed "hot" fusion, we heat atoms until they have about

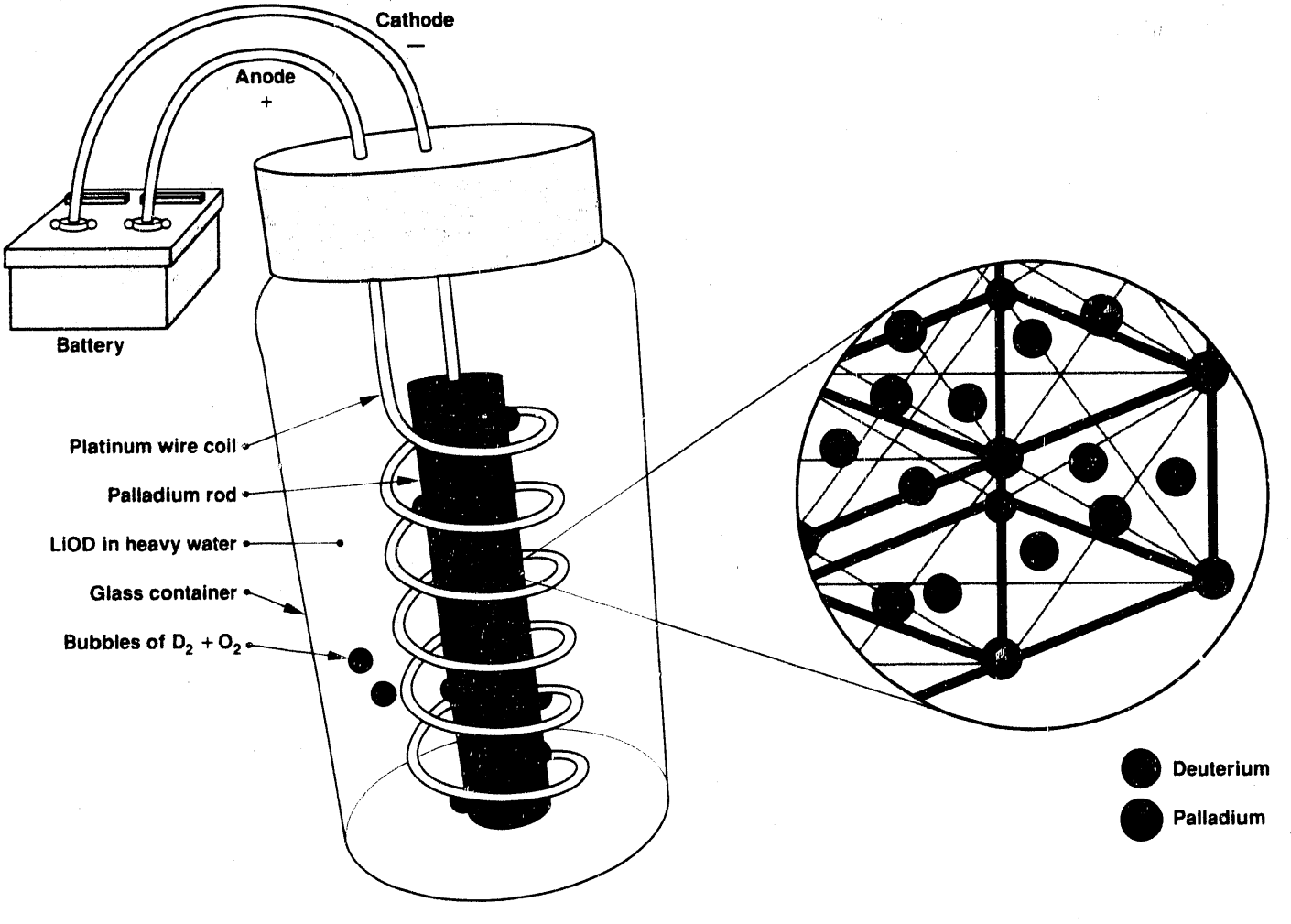


Figure 1. *Setup for the Fleischmann and Pons experiment. A palladium electrode wrapped with a coil of platinum was immersed in a beaker containing lithium salts and heavy water (consisting of oxygen and the mass-2 isotope of hydrogen called deuterium). After running an electric current through the device for several weeks, the palladium began to produce heat, according to these researchers, releasing about four times more power than was put into the setup. Their reasoning was that ions of deuterium migrated into the crystal lattice of palladium (see enlarged area), where they became so tightly packed that they fused to form atoms of helium.*

5 keV or about 50 million degrees of energy. Such heating allows atom pairs to approach each other closely enough to fuse before electrostatic repulsion pushes them apart. At this temperature and at typical fusion-reactor fuel densities (10^{14} atoms/cm^3), the reaction rate of D–D fusion is 10^{-5} reactions per second. If we use the value given by Fleischmann and Pons of 26 W/cm^3 of heat produced in their electrolytic cells, then the reaction rate for D–D fusion—if it were indeed the source of heat—would be about 10^{-9} reactions per second, much too high for consistency with the early reported neutron measurements according to our current understanding of D–D fusion.

Fleischmann and Pons conjectured that D–D fusion reactions of a rare type, producing ^{4}He (ordinary helium nuclei), were taking place at room temperature—thus the term "cold" fusion—and were responsible for the reported excess heat in their experiments.[2] This type of reaction is known to occur with low probability in a hot gas, where it produces a high-energy gamma ray but no neutrons. They also speculated on the possibility of a fourth reaction in which the energy is transferred directly to the crystal lattice (see cover). They postulated that if their heavy-water electrolytic cell, which included a palladium cathode, was run at a sufficient overpotential, the deuterium atoms driven into the palladium would be under the intense pressure of 10^{26} atmospheres. This large so-called "confining pressure" would then enable fusion reactions to take place. In fact, the interpretation of an equivalence between the overpotential and a high pressure is false. Nevertheless, the fact that the purported heat release per D–D pair far exceeded what could come from a chemical reaction (limited to a few electron volts per pair) fueled their speculation that fusion was responsible for the results.

Conflicting Claims

The ability of palladium metal to absorb hydrogen and hydrogen isotopes and the structure of palladium when fully loaded with deuterium are well-known. The 0.17-nm spacing between adjacent deuterium atoms is more than twice the spacing in a gas of D_2 molecules. Our current understanding of nuclear fusion indicates that a distance ten times smaller is needed for fusion.

Nontheless, the slim chance that the Fleischmann and Pons experiments were being interpreted properly was tempered by the near-infinite benefit that could be derived if the experimenters were proven to be correct. Thus, the scientific effort that took place during the next few weeks (see the timetable on p. 4) was rivaled in recent scientific history only by the activity created by the discovery of high-temperature superconductivity. Many scientists made analogies between the two situations but, unlike the latter discovery, reproduction of the Fleischmann and Pons results was the exception rather than the rule. Most major laboratories around the world found no excess heat or evidence of nuclear processes. The few researchers who confirmed the observation of excess heat, such as those at Texas A&M University,[3] usually reported excess heat values in the range of 10 to 20%. Similarly, the few groups that found evidence for neutron emission, such as researchers at the Los Alamos National Laboratory[4] and Brigham Young University,[5] reported levels so close to background that detection methods quickly became the center of attention.

Because the heat excess over the input power claimed by a few groups was in the 10 to 20% range, it quickly became obvious that the calorimetry used in cold fusion experiments needed to be improved. During the electrolysis process, a fraction of the input power is used to separate D_2O into the D_2 and O_2 gases that essentially escape from an open system. This fraction, which exceeded the excess heat in most cases, can be calculated by multiplying the current times 1.527 volts. Because very few of the experiments, and none of the early ones, used closed calorimetry, this amount of power was subtracted from the product of the input voltage times current to determine how much of the power was actually going into a cell. Experimenters then calculated the possible excess heat by comparing the difference between that reduced power and the output heat, which was determined from calorimetry measurements of various kinds.

Great care is needed in making heat measurements of this kind because the sources of potential error are numerous and difficult to quantify. Fluctuations of parameters in the cell, long-term drifts, calibration accuracy, and a host of other phenomena are important in the interpretation of results. Herein lies the controversy: is the claim for *any* excess heat actually proven? Except for the first claims of Fleischmann and Pons and those made at the Santa Fe conference on cold fusion by Robert Huggins[6] from Stanford University, the amounts of excess heat reported by all supportive experiments were at levels well below the power needed to separate the gases that presumably boil off. Early support for the claim of excess heat came primarily from the group at Stanford University and two groups at Texas A&M University. Negative results were reported by a long list of researchers throughout the world and at LLNL.[7]

Fleischmann and Pons noted that their low neutron count, which was about nine or ten orders of magnitude too low for conventional D–D fusion, represented a

A Timetable of Key Claims Related to Cold Fusion

1926 Fritz Paneth and Kurt Peters in Berlin first claim to have observed the fusion of hydrogen under pressure to form helium in finely divided palladium metal. It was already well known at the time that certain metals such as palladium can absorb large quantities of hydrogen. In a short note immediately following this claim, *Nature* issued the statement: "This announcement, if correct, is of great importance and will evoke even more interest than the claim by Miethe and Stammereich to have transmuted mercury into gold . . . Belief or disbelief in the . . . message must be reserved pending further and more definite evidence." After substantial criticisms and further studies, the two researchers withdrew the claim of helium synthesis.

1927 The Swedish scientist John Tanberg proposes using electrolysis to force hydrogen into palladium metal. After obtaining heavy water from Neils Bohr in 1932, he filled palladium rods with deuterium by electrolysis and then applied a large electric current to heat the palladium. He warned coworkers to go home during the experiments after calculating that all the deuterium would be equivalent to 1000 kg of dynamite, if exploded. He observed no effect.

March 23, 1989 Martin Fleischmann and Stanley Pons working at the University of Utah publicly announce at a press conference the initiation of nuclear fusion (the production of excess steady-state heat, low-level neutrons, gamma rays, and tritium) in simple electrochemical cells of a palladium cathode surrounded by a spiral platinum anode immersed in a bath of heavy water (D_2O) as the electrolyte.

March 24, 1989 Steven Jones and coworkers at Brigham Young University report at a press conference the detection of neutrons with energy characteristic of a fusion reaction in an electrochemical cell containing deuterium and titanium electrodes.

April 11, 1989 C. R. Martin, B. E. Gammon, and K. N. Marsh at Texas A&M University announce confirmation of excess heat from isothermal heat-leak calorimeter measurements with an open cell.

April 18, 1989 Researchers at the Frascati Center (Ente Nazionale Energie Alternative) in Italy, led by F. Scaramuzzi, obtain provisional results of apparently high statistical significance, suggesting fusion as a dynamic effect by varying pressure and temperature in a titanium-deuterium system.

Late April 1989 At John Bockris' laboratory at Texas A&M University, tritium appears in six different electrochemical cells during one week.

May 23–25, 1989 Both positive and negative results on excess heat are described at the Workshop on Cold Fusion Phenomena, Santa Fe, New Mexico, sponsored by the Los Alamos National Laboratory.

November 1989 The U.S. DOE Energy Research Advisory Board issues its final report, *Cold Fusion Research*. The Cold Fusion Panel concludes that ". . . the experimental results of excess energy from calorimetric cells reported to date do not present convincing evidence that useful sources of energy will result from the phenomena attributed to cold fusion." In addition, the Panel states that ". . . the present evidence for the discovery of a new nuclear process termed cold fusion is not persuasive."

March 1990 Proponents at the First International Cold Fusion Conference suggest that the observed emissions of tritium are the ". . . unassailable signature of a nuclear reaction."

To date, hundreds of experiments have been conducted around the world involving thousands of hours of research. Most investigators find no effect, while a few others report positive results. Many positive claims made in the past have been withdrawn.

discrepancy in their data. To understand why this was true, we must examine the reaction outcomes, shown schematically in Figure 2, when two deuterium atoms fuse. (These reactions are described in equation form in the next article.) Two dominant D–D reactions occur with nearly equal probability. One of these reactions yields tritium and a 3.02-MeV proton, while the other yields the mass-3 light isotope of helium, ^{3}He, and a 2.45-MeV neutron. A third reaction with much lower probability produces ordinary helium, ^{4}He, and a high-energy (23.85-MeV) gamma ray. Thus, each watt of excess heat from cold fusion should have been accompanied by about 10^{12} atoms/s of tritium or ^{3}He, with their attendant protons and neutrons. Fleischmann and Pons, who clearly recognized the problem, assumed that the normal D–D reactions were suppressed at room temperatures and that the rare branch of the D–D reaction producing ^{4}He was responsible for the heat. Because 23.85-MeV gamma rays were not observed, this reaction would have required that these high-energy rays also be absorbed or suppressed by some means. However, there is no theoretical reason to believe that the ratio of the two dominant reactions (the branching ratio) would change from about 50:50 at ordinary fusion temperatures (several to tens of keV) to very different values at low energy. For example, the results for muon-catalyzed fusion at room temperature are consistent with the normal branching ratio. The inconsistent neutron output was the major cause for initial skepticism about cold fusion.

The limit of detection established by the best neutron detectors is about

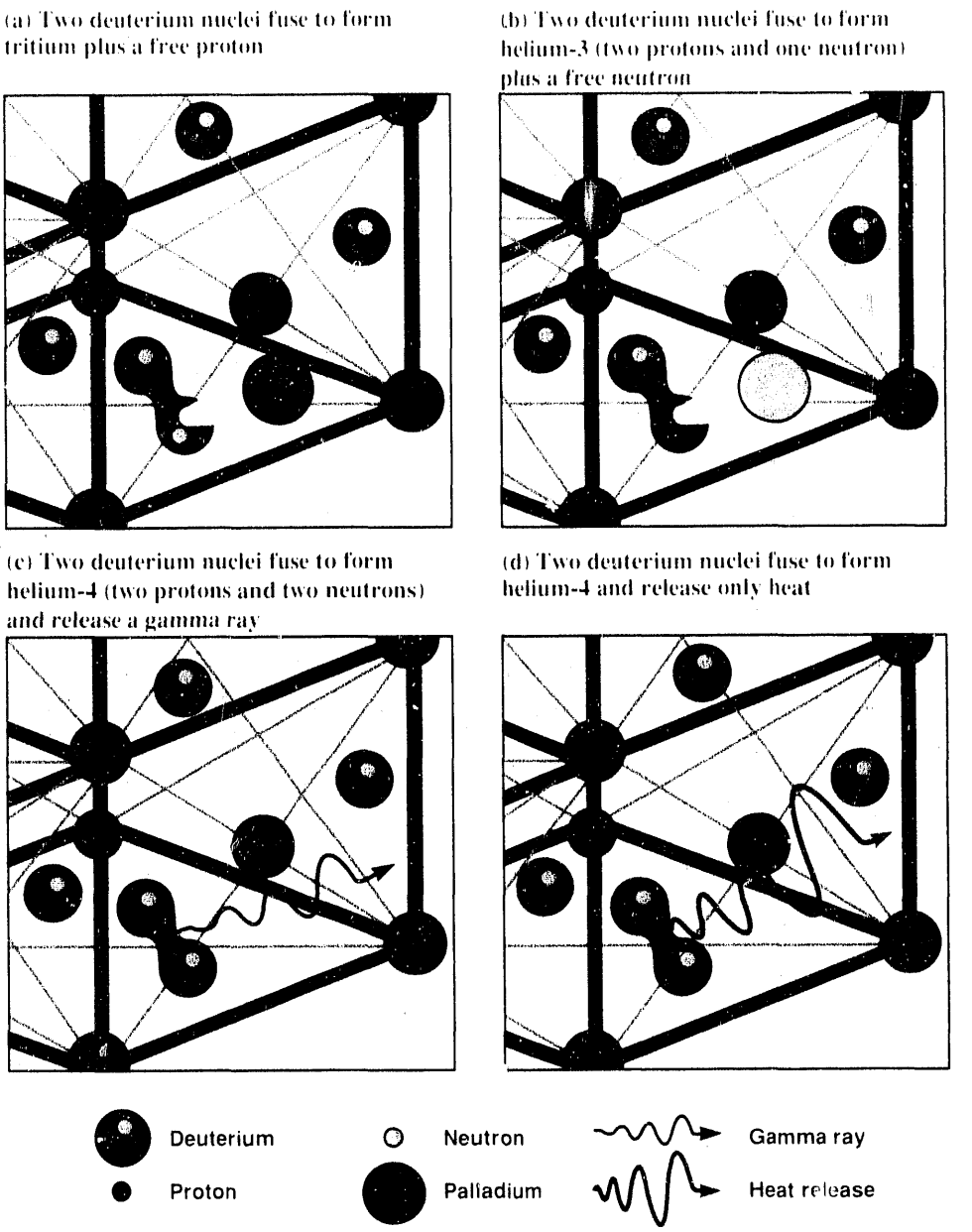


Figure 2. *Three reactions (a–c) are known to be possible in a hot gas when two deuterium atoms fuse. These drawings are similar to the enlarged area of Figure 1 in that they show deuterium within the crystal lattice of palladium. Researchers used sensitive detectors to look for the products of these reactions. For the dominant reactions (a and b), they looked for tritium and ^{3}He remaining in the lattice and for prompt neutron flux. Reaction (c) is far less likely than the two dominant ones, although its gamma rays should be observable. A fourth reaction (d) has been proposed as a possible mechanism to explain the lack of observed radiation in the cold fusion experiments. None of these reactions is expected to occur in a palladium lattice, however, given the large known spacings of the lattice sites.*

10^{-24} reactions per second per D–D pair in deuterium-saturated palladium, and this was about the rate claimed by cold fusion experimenters.* (Recall that the fusion rate in a D–D gas at room temperature is 10^{-74} reactions per second per pair.) That the reaction rate for the new phenomenon should increase by more than 50 orders of magnitude to a rate almost precisely at the limit of detection appeared to be a result of the most fortuitous nature to much of the scientific community. In addition, to account for the fact that many observers were unable to replicate the results, the supporters of cold fusion noted that some cells worked, others did not, and they could not explain why.

If the dominant fusion reactions were taking place in the electrolytic cells, then the generation of helium and tritium *in situ* in the palladium rods at rates consistent with heat production would have left a signature easily identified by materials analysis. Both helium and some tritium would remain trapped in the rod. Using sensitive analysis equipment, noble-gas mass spectroscopy, we found neither helium nor tritium above detection limits in a palladium rod supplied by Srinivasan, an experimenter in one of the Texas A&M groups claiming an excess of heat from their device. When we analyzed a sample of palladium wire provided by Fleischmann and Pons in a double-blind experiment involving several other laboratories, our results were similarly discouraging.

At the Santa Fe cold fusion workshop, sponsored by Los Alamos National Laboratory in May 1989, two groups at Texas A&M reported that tritium had accumulated in their cells. These results were confirmed by independent and competent analysis at Los Alamos. Recently, however, one of these groups, headed by Kevin Wolf, reported that their palladium rod was contaminated with tritium when it had been received from the supplier. Results from the second group are under scrutiny, and the June 15, 1990, issue of *Science* contained an article suggesting the possibility of intentional spiking with tritium. No other researchers have reported reproducible accumulations of excess tritium in electrolytic cells by mechanisms associated with cold fusion.

In April 1989, a group from the Frascati Ente Nazionale Energie Alternative in Italy reported large bursts of neutrons from a "dry fusion" experiment.[8] This announcement opened a new area of investigation. For their dry fusion work, these researchers used titanium shavings immersed in deuterium gas at 60 atmospheres of pressure and warmed from liquid-nitrogen temperatures (−197°C) to room temperature to produce the bursts. This result stimulated much discussion and seemed to be related to 1986 work in the Soviet Union.[9] The Soviet experiment accelerated a lithium deuteride pellet and slammed it into a target. On fracturing, the pellet produced a burst of neutrons, according to the published paper. The Soviet experimenters conjectured that high electric fields were generated in the process of fracturing (they observed x rays up to 4 MeV), accelerating the deuterium ions to energies needed for fusion. Speculation that a thermal fracturing process was at work in the burst experiment arose from the Soviet results.

Experimental results similar to those from the Italian work, but with lower neutron levels, were reported by Los Alamos. In an exhaustive set of experiments conducted at LLNL, culminating in the use of D–T gas, as described in the next article, much of what we saw could be attributed to errors in interpretation of the detector signals. We were able to place an upper bound on the number of neutrons that could have resulted from this process, and this bound was far lower than that suggested by other groups. The Frascati researchers have since disclaimed their original results due to failures of replication.

False Hope

During the months when cold fusion activity was at its peak, there were many instances when preliminary results of an encouraging nature were later retracted, often within a short time. Retractions were usually the result of finding errors in the interpretation of data, such as thermal drift in neutron detectors that was misinterpreted as real neutron counts. In retrospect, such outcomes were one consequence of the intense interest generated by the press and the substantial pressure to give out bits of information as soon as observations were made. Although such demands for immediate information may not be conducive to good science, in the long run the scientific method once again emerged as our best tool for differentiating among early claims and pointing the way to fact.

Even though the phenomenon of cold fusion turned out to be a false hope, the search itself had redeeming aspects. We obtained a better understanding of instrumentation errors in searching for nuclear products generated at extremely low rates over hours or days, a regime that is not normally explored. The

*The early result of 10^{-24} was shown to be in error.

difficulties of calorimetry, the complexity of palladium hydrides, and the fundamental nuclear reaction rates at low temperatures were all fascinating subjects that had to be addressed. In addition, the quest for cold fusion involved a broad range of disciplines that resulted in new collaborations among people from diverse areas in each of the participating institutions. Scientists at LLNL had the opportunity to apply a diversity of talent to a problem that captivated the public interest and required a competent and rapid response.

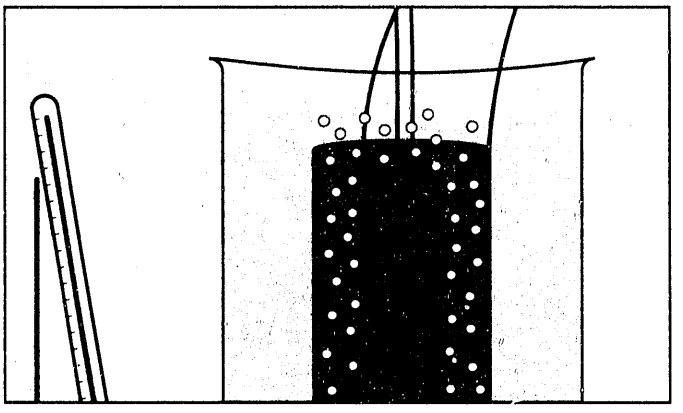
Key Words: deuterium-deuterium fusion; cold fusion; palladium hydride.

References

1. J. Rafelski, M. Gajda, and D. Harley, *Theoretical Limits on Cold Fusion in Condensed Matter*, University of Arizona, Tucson, Arizona, Rept. AZPH-TH/89-19 (1989).
2. M. Fleischmann and S. Pons, "Electrochemically Induced Nuclear Fusion of Deuterium," *J. Electroanal. Chem.* **261**, 301–308 (1989).
3. S. Srinivasan et al., "Evidence for Excess Heat Generation Rates During Electrolysis of D_2O in LiOD using a Palladium Cathode," presented at the Workshop on Cold Fusion Phenomena, Santa Fe, New Mexico (May 23–25, 1989).
4. H. E. Menlov et al., paper submitted to *Nature*, 13 October 1989.
5. S. E. Jones et al., "Observation of Cold Nuclear Fusion in Condensed Matter," *Nature* **338**, 737–740 (1989).
6. A. Belzer, U. Bischler, S. Crouch-Baker, T. M. Gur, M. Schreiber, and R. A. Huggins, "Two Fast Mixed-Conductor Systems: Deuterium and Hydrogen in Palladium," paper presented at the Workshop on Cold Fusion Phenomena, Santa Fe, New Mexico (May 23–25, 1989).
7. K. I. Thomassen and J. F. Holzrichter, Eds., *LLNL Research on Cold Fusion*, LLNL Rept. UCID-21809 (1989).
8. A. De Ninno et al., "Evidence of Emission of Neutrons from a Titanium-Deuterium System," *Europhys. Lett.* **9** (3), 221–224 (1989).
9. V. A. Kluyev et al., *Sov. Tech. Phys. Lett.* **12**, 551 (1987).

For further information contact Keith I. Thomassen (415) 422-9815.

LLNL Experiments on Cold Fusion



Our studies on cold fusion yield no evidence of significant nuclear fusion reactions that would support recent claims in the literature. Instead, they reveal that various artifacts and spurious results can arise even in apparently simple experiments.

In March 1989, scientists working at the University of Utah publicly announced the attainment of cold fusion in simple electrochemical cells. If confirmed, these and subsequent positive findings would have been of enormous scientific interest and economic value. For example, nuclear fusion observed at room temperature and under the conditions described by the Utah investigators would seriously question our understanding of the fundamental nuclear reaction processes gained over the last half century and would require the development of entirely new descriptions for such processes. The potential economic payoff of applications based on cold fusion—the idea of cheap and abundant energy from seawater—would be spectacular indeed.

After the early announcements were made to the press and before articles began to appear in the scientific literature, many research groups associated with universities and with national and industrial laboratories began participating in the quest to confirm the reports of cold fusion. At LLNL, independent teams of scientists were quickly formed in efforts to reproduce and extend the experiments reported by the media. The first experiments were done on a small scale. At the request of the Secretary of Energy (a month later), a more substantial effort was subsequently initiated. We first attempted to replicate the electrochemical cell experiments using the incomplete information available from the Utah researchers and then extended our experiments to place upper bounds on the generation of heat and the production of nuclear products. In a collaborative effort with scientists at Texas A&M University, we analyzed electrodes and electrolytes for fusion products in a cell producing 10% excess heat (no such products were found). We participated in double-blind experiments with Batelle Pacific Northwest Laboratory in which we analyzed palladium material from electrolytic cells used by researchers at the University of Utah. We also attempted to replicate the experiment reported by the Frascati group in Italy that first found neutron bursts when high-pressure deuterium gas in a cylinder containing titanium chips was temperature-cycled.

Here we briefly describe some of the fundamental principles that characterize nuclear fusion reactions as we understand them today and

discuss some major problems associated with claims of cold fusion. We then review several of our studies that can help to explain the phenomena or artifacts underlying "desk-top" fusion results.

Fusion Reactions

Nuclear fusion reactions involve the joining of light nuclei to form heavier nuclei and are accompanied by the release of energy. This energy is manifested in the form of the kinetic energy of the heavier product nuclei and of the neutrons, protons, and gamma rays that are also produced in the reactions. In the case of the fusion of two deuterium nuclei, which are hydrogen nuclei with an atomic mass of 2, three outcomes of the are known to occur in a hot gas. The first two reaction outcomes are equally likely:

$$D + D \rightarrow {}^{3}He + n + 3.27\ MeV\ , \quad (1)$$

and

$$D + D \rightarrow {}^{3}T + p + 4.03\ MeV\ . \quad (2)$$

In these reactions, D is deuterium, ^{3}He is the mass-3 light isotope of helium, ^{3}T is tritium (the mass-3 isotope of hydrogen), n represents a neutron, and p represents a proton. In addition, it is also possible, but much less likely, that the outcome will be

$$D + D \rightarrow {}^{4}He + \gamma + 23.85\ MeV\ . \quad (3)$$

Here, ^{4}He is an ordinary helium nucleus and γ represent a high-energy gamma ray.

Although fusion reactions release large amounts of energy when they occur, it has nevertheless been found that a considerable amount of energy must be supplied up front to cause them to take place. The fusion of two nuclei takes place under the influence of the so-called strong nuclear force. Although powerful, this attractive force only acts over an extremely short distance. Consequently, two nuclei must be brought very close together for the strong nuclear force to take over and produce fusion. At the same time, however, the electromagnetic force, in the form of the Coulomb interaction, causes the positively charged nuclei to repel each other. Because the Coulomb repulsion acts over a much greater distance than does the strong nuclear force, it is necessary to do work against it to bring the nuclei close enough together for the strong nuclear attractive force to become effective and produce fusion.

In the 1930s, the first fusion reactions produced in the laboratory were brought about by using particle accelerators to speed up some nuclei and cause them to collide with others at high energies. In the case of D–D fusion, energies at least on the order of about 10 keV are necessary to produce a significant reaction rate by this method. This was the operating principle of the Rotating Target Neutron Source at LLNL, which produced D–T fusion reactions. Although it was the most intense continuous source of fusion neutrons in the world, it was not a producer of net energy, since only a small fraction of accelerated nuclei undergo fusion in this approach (the rest interact with electrons and dissipate their energy as waste heat).

The only known method for obtaining net energy from nuclear fusion is the thermonuclear approach. In this approach, a relatively large number of atoms are brought together and are heated to temperatures in the neighborhood of 100 million degrees (10 keV). At such temperatures, the atoms become ionized and the nuclei travel at sufficiently high speeds that, upon collision with each other, some (principally those in the high-velocity tail of the statistical distribution) can overcome the Coulomb repulsion and approach one another so closely that the strong nuclear force can produce fusion. This is the principle at work in the sun and other stars, in thermonuclear explosive devices, and in magnetic and laser fusion.

In all known cases, either energetic accelerated particles or high temperatures are necessary to produce nuclear fusion reactions. In the context of recent discussions, the only fusion known has been "hot" fusion. The term "cold" fusion has been applied to experimental approaches recently claimed to produce significant rates of nuclear fusion reactions at temperatures near room temperature and/or with applied energies of no greater than a few electron volts. Such approaches have included the use of electrochemical cells, temperature cycling of pressurized gas-metal systems, fracturing of crystals, ion implantation, and the application of pulsed electrical current to multiple alternated layers of metal and semiconductor in the presence of gas.

If D–D fusion were to occur at significant rates in a cold fusion experiment, it would be manifested in three ways:

1. Observable energy would show up in the form of heat resulting from collisions of the recoiling heavier product nuclei and protons with surrounding matter.
2. Energetic particles (neutrons, gamma rays, protons, tritons, and x rays) would appear in observable numbers.
3. Stable products of fusion (^{3}He, tritium, and smaller amounts of ^{4}He) would remain.

The ratio between the amounts of heat and fusion products would be

expected to correspond to what is produced by the known D–D fusion reactions. In addition, the relative amounts of the various fusion products would be expected to agree with the known branching ratios, which describe the relative likelihoods of the three different reaction outcomes.

Experiments Using Electrochemical Cells

The first claims for the observation of cold fusion were based on experiments with electrochemical cells. As shown in Figure 1 in the preceding article (p. 2), a basic electrochemical cell consists of two conducting electrodes placed in contact with a conducting electrolyte and connected to an external electrical circuit with a power supply. In the cells that purportedly produced excess heat as well as neutrons, gamma rays, and tritium from cold fusion, the electrodes consisted of a palladium metal cathode (negatively polarized), a platinum metal anode (positively polarized), and a conducting solution of lithium deuteroxide (LiOD) dissolved in heavy water (D_2O). With a few volts dc applied, such a cell will electrolyze the heavy water—that is, break it up into D_2 gas, which bubbles out at the cathode, and O_2 gas, which bubbles out at the anode. In the course of this electrolysis, some deuterium atoms will also diffuse into the palladium, which has been known for a long time to have high solubility and high diffusivity for hydrogen isotopes. The original proponents of cold fusion were apparently under the impression that these circumstances might allow the deuterium nuclei to approach each other sufficiently close that fusion could occur. They claimed to have made observations that supported this hypothesis.

Two major problems are associated with these claims. First, it is difficult to understand how the strong Coulomb repulsion between two deuterium nuclei could be overcome in such a system. Second, the amount of excess heat purportedly observed was many orders of magnitude larger than what would have been expected from the number of neutrons and gamma rays claimed to have been detected if nuclear fusion were responsible for both results. Thus, if "cold fusion" was occurring, it was not consistent with current theory. If it were indeed real, then cold fusion would not only be a major breakthrough for practical energy production, but it would also require a revolution in nuclear and solid-state physics theory.

One of the first groups at LLNL to begin experiments testing the validity of claims for cold fusion was a collaboration of scientists from the Chemistry and Materials Science Department, the Magnetic Fusion Program, and the Laser Fusion Program. We initially attempted to reproduce the electrochemical experiments reported in the media, using the sketchy information available about the details of the experiments. As more information became available, we redesigned and altered the focus of subsequent experiments.

The main thrust of our work became the matched-cell differential thermometry experiment (Figure 1). An important feature of this experiment is that we used two cells—one incorporating LiOD in heavy water as the electrolyte and the second containing LiOH in ordinary water as a control. We carried out electrolysis in both cells, monitored the temperatures inside the helically wound palladium wire cathode of each cell, and monitored neutrons. After several days of operation, we analyzed samples of the palladium for ^3He and ^4He by vacuum fusion mass spectrometry. This technique involves melting the palladium and analyzing the gas atoms evolved by first ionizing them, then accelerating the ions, and finally passing them through a magnetic field to see how much their trajectories are deflected (the various isotopes are deflected by different but distinct amounts and thus can be identified).

The significance of our experiments is that they included control cells, which are necessary to rule out experimental artifacts, and that they sought to detect the stable products of fusion. The degree of precision possible in detecting excess heat by our experiments was not high in comparison to the much more difficult, costly, and time-consuming method of closed-cell absolute calorimetry. However, the precision was more than adequate to detect the amounts of heat reported by the original cold fusion researchers. Likewise, our neutron monitoring was of low precision, conducted primarily as a radiation-safety precaution. Nevertheless, such monitoring was adequate to detect any neutron output that would be of practical significance. Later experiments were designed to detect neutrons using specially built detectors with higher detection efficiency and lower background count rate.

We found no evidence for cold fusion in any of the LLNL experiments using electrochemical cells. We did, however, observe several artifacts. One interesting artifact arose from the temperature sensitivity of neutron detectors. We used a BF_3 Bonner sphere-type neutron survey meter placed about 1 m from the cells. A rise in apparent

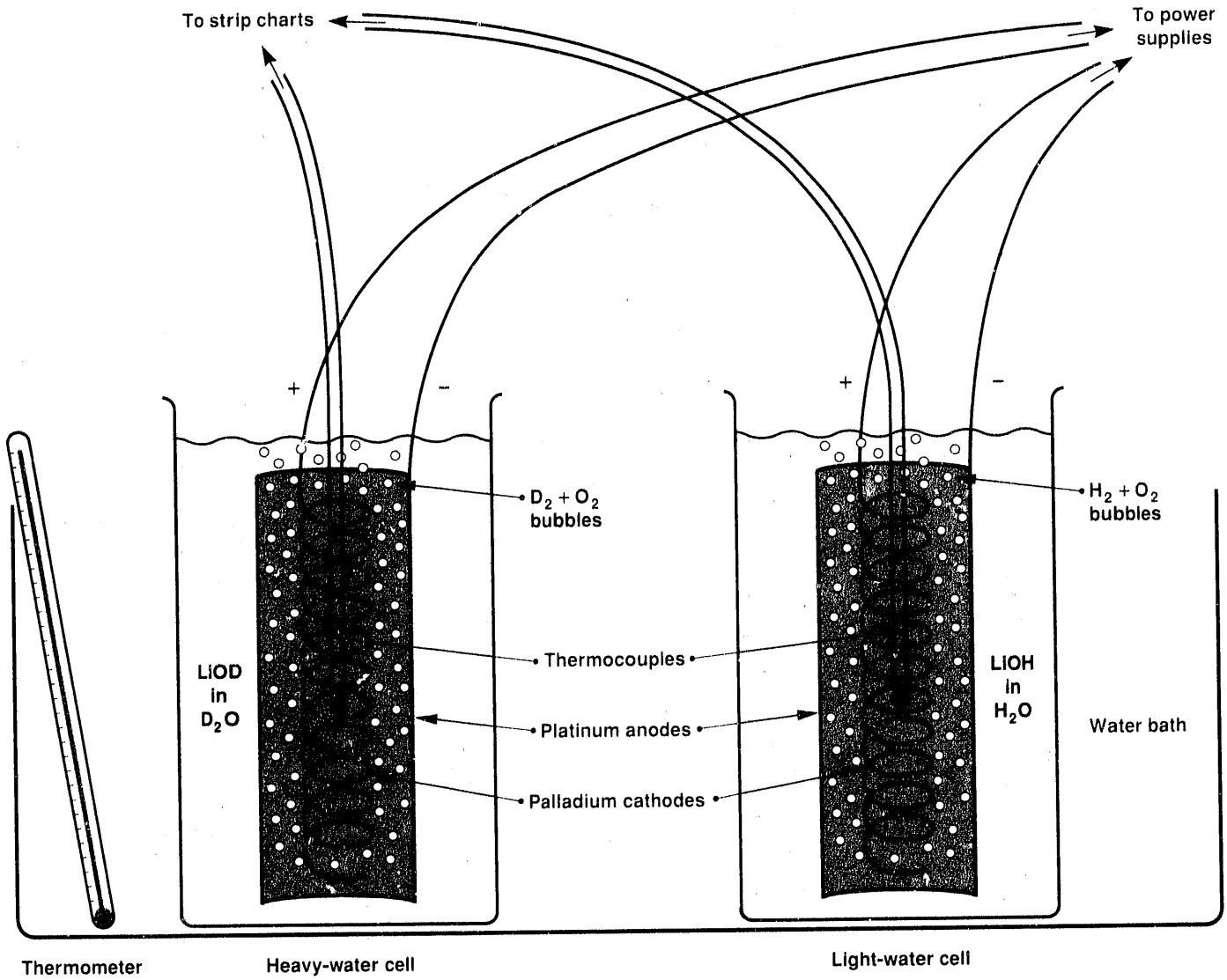


Figure 1. *Setup for matched-cell differential thermometry experiments at LLNL. Our concept was to fabricate two nearly identical cells, using the light-water cell as a control for purely chemical effects. Each palladium cathode was made from 1-mm-diam wire wound in the shape of a helix. Each anode was made of platinum foil wrapped concentrically with the cathode into a cylinder about 2 cm in diameter. A metal-sheathed thermocouple was inserted into a closed-end glass tube inside each helical cathode for temperature monitoring, and the output was fed to a dual-input strip chart. Identical current-controlled power supplies powered the two cells. The cells were first allowed to reach steady-state conditions at zero input current, and then the current through both cells was slowly raised over various time intervals (up to 414 hours in one of our three experiments). We then removed the palladium wires for surface analysis and for ^3He and ^4He analysis by vacuum fusion mass spectrometry. We found no significant change in the temperature of either cell once steady-state operation was achieved at each current, and the neutron count rate monitored by a sphere-type neutron survey meter remained constant in two of our three experiments. A rise in apparent neutron-counting rate in our third experiment was coincident with failure of the building air conditioning system and could be attributed to a temperature rise in the survey meter. Two new neutron detectors with improved efficiency and lower background noise, which were specially designed and built for our experiments, also showed no neutron signal above detection limits.*

neutron-counting rate in one of our experiments was coincident with a significant rise in room temperature that resulted from failure of the building air conditioning system. When the air conditioning came back on, the neutron counting rate dropped back to the original background value. Thus, the rise in signal was attributed to a temperature increase in the survey meter. The use of specially designed neutron detectors with improved efficiency and lower background count rate in later experiments confirmed no neutron signal above the detection limits.

Another artifact arose after removing the gas-charged palladium cathode from the cells and exposing them to air. We found that the wires heated to incandescence and burned the paper and plastic bags on which they were resting. After a few minutes, the wires and samples cooled to room temperature. Such heating to incandescence can be readily explained as an avalanche process. When the electrochemical overpotential was removed, excess deuterium (hydrogen) began to evolve from the palladium. On arrival at the palladium surface, the deuterium (hydrogen) was catalytically oxidized to D_2O (H_2O) with oxygen from the air. The oxidation produced heat, raising the temperature of the wires and the diffusion coefficient of deuterium (hydrogen), and lowering the solubility of deuterium (hydrogen) in palladium, eventually heating the wire to red heat. Once the deuterium (hydrogen) supply was exhausted, the palladium cooled again. Upon termination of one experiment, we quickly dried one of the palladium cathodes and placed it into an argon atmosphere while monitoring the temperature with a thermocouple. Without oxygen present, we found no production of heat due to oxidation. We communicated what we had observed informally to other groups, who found this helpful in interpreting their own results.

Double-Blind Experiments

When cold fusion was first reported by the press, members of the Nuclear Chemistry Division at LLNL recognized that measurements of helium, tritium, and neutron or proton activation products would be useful for diagnosing cold fusion reactions. Thus, we became involved in a series of experiments in which we measured helium in palladium wire, tritium in fusion-cell electrolyte, and the gamma-ray flux from palladium wire, especially looking for gamma rays from ^{105}Ag [an anticipated product of (p,γ) reactions on palladium initiated by protons from one of the principal D–D reaction branches]. All of our results were negative.[1]

Perhaps our most significant negative result came from an analysis of a sample supplied by researchers from Texas A&M University. We determined that both helium and tritium levels were very low from a cell purported to have produced significant excess heat. Our results indicated that the amounts of ^3He, ^4He, and ^3H were, in fact, many orders of magnitude below levels corresponding to the excess heat that was reportedly produced.

In our final efforts, we participated in double-blind experiments that were overseen by Pacific Northwest Laboratory (PNL). In this work, helium analyses were performed on palladium rod that had been obtained from Fleischmann and Pons at the University of Utah. This work involved a consortium of six laboratories:

- LLNL.
- Rockwell International Energy Technology Engineering Center.
- University of California, Santa Barbara.
- Delft University of Technology.
- Woods Hole Oceanographic Institution.
- Rockwell International, Rocketdyne Division.

For the double-blind experiments, Fleischmann and Pons prepared five samples that were identified only by a letter of the alphabet when received by each participating laboratory. No information regarding sample history was provided; neither was any information supplied regarding potential sample history. Thus, each lab received truly unknown samples. The identity of a given sample could be revealed only by combining information made available to PNL and the University of Utah.

On October 6, 1989, PNL and the University of Utah exchanged information, PNL supplying the helium abundances and the University of Utah providing the cell descriptions (see Table 1). The samples proved to be highly variable in helium content, and subsamples analyzed within individual laboratories also showed large variations. No ^3He was detected in any sample ($<5 \times 10^{-17}$ gram-atoms/cm^3 palladium). For the units used in Table 1 (nanogram-atoms ^4He/cm^3 palladium), the expected value for the implanted samples is approximately 970, equivalent to an apparent excess heat production of 10%. The equivalent excess heat production for sample 5 was less than 0.01%. That is, we demonstrated a capability to easily find helium over the dynamic range required. This limit would have been much lower had the initial rod not contained such a large initial amount

of helium. Subsequent measurements indicated that the contaminating helium was located near the surface of the rod.

On November 7, 1989, Pons reported to PNL that rod 5 had generated on the order of 5–8 mW continuously for 24 days. Although this rod had produced much less heat than anticipated, Pons and Fleischmann elected to include it instead of one that produced more excess heat because they felt the replacement would not be consistent with the terms of the double-blind experiment. This level of excess heat production (0.1%) is so small that it cannot reliably be distinguished (within experimental error) from zero.

Given the initial helium contamination of the palladium rod, the variability of the helium implantation procedure, and the failure of cells provided by the University of Utah to produce heat, little can be said regarding cold fusion phenomena from this experiment. (The study does confirm the fact that helium is reasonably well retained in palladium during electrolysis.) Indeed, the results of all of our radiochemical measurements were consistent with the conclusion that no fusion reaction occurs within so-called cold-fusion cells. In addition, the detection limits for our radiochemical methods were excellent, and we conclude that no cell examined to date came within many orders of magnitude of the original results reported by Fleischmann and Pons.

Metal Hydride Systems under High Gas Pressure

Another kind of experiment that seemed to support the idea that fusion could be enhanced when deuterium is absorbed by a metal was initially performed at the Frascati laboratory in Italy. These experiments generally used titanium rather than palladium as the host material. Later, positive results were also reported from the Los Alamos National Laboratory and the National Cold Fusion Institute, Utah.

The researchers at Frascati questioned whether electrolysis and palladium must be fundamental to the cold fusion process. The substitution of titanium for palladium seemed to be a potentially fruitful alternative. The titanium and palladium lattices have unit cells of similar dimensions; however, titanium can absorb twice as much hydrogen as palladium. To simulate the nonequilibrium conditions that seemed to be essential in the electrolytic catalysis, titanium was pressurized by exposure to 5×10^6 Pa of D_2 gas, cooled with liquid nitrogen to −197°C, and then allowed to warm to room temperature. The purpose of this procedure was to stress the titanium sample by subjecting it to high pressure and extremes of temperature. Under these conditions, a neutron detector showed short bursts of up to 20 counts. After corrections were made for detection efficiency, the implication was that nearly 20,000 fusions had occurred during each burst. When the deuterium gas was removed from a similar sample during later studies, the detector count rate began to rise dramatically, peaking after 10 hours at a level corresponding to nearly 5000 fusions per second. Results reported from Los Alamos were obtained under similar conditions, but the corresponding fusion rates were 1000 to 10,000 times smaller than those observed at Frascati.

The researchers themselves recognized that many questions were posed by their studies. For example, what was the pressure and temperature in the cell when an event was observed? How much deuterium was absorbed by the titanium? Was the metallurgical state of titanium important? Because the time scale could be an important clue in

Table 1. Results of helium analyses for the Fleischmann and Pons palladium rods.

Palladium rod preparation	Helium analysis, ng-atoms $^4He/cm^3$ palladium	
	Range of reported values	LLNL value
Rod 1. Ion-implanted with 3×10^{-7} mol ^4He (500 keV), then electrolyzed at 800 mA in 0.1-*M* LiOD for 28 days	236–1303	790
Rod 2. Original palladium wire as received from the manufacturer, Johnson and Matthey	0.04–2.0	0.20
Rod 3. Ion-implanted with 3×10^{-7} mol ^4He (500 keV) and otherwise untreated	5.3–309	11.0
Rod 4. Ion-implanted with 3×10^{-7} mol ^4He (500 keV), then electrolyzed at 800 mA in 0.1-*M* LiOH for 28 days	120–775	420
Rod 5. Electrolyzed at 800 mA in 0.1-*M* LiOD for 28 days	0.16–1.04	0.63

understanding the phenomenon being observed, how long did the bursts last? Most fundamentally, did the signals represent convincing evidence for the production of 2.5-MeV neutrons from D–D fusion?

Although such questions were easy to formulate, they were difficult to answer. To introduce temperature and pressure sensors in the setup, holes would have to be made in the wall of a high-pressure reaction cell, and sensors welded in place. However, such modifications introduce the possibility of a violent explosion should any of the welds fail. For this reason, experiments with instrumented high-pressure vessels are usually carried out in shielded vaults. For safety and convenience, early experimenters made sample containers from small high-pressure bottles similar, except in size, to pressurized gas cylinders. These bottles, however, did not include pressure and temperature sensors.

The neutron detectors used at Frascati and Los Alamos also made it difficult to extract any information about the particles being measured from the observed signal. The active element of both detectors was only sensitive to neutron energies around a few thousand electron volts. The use of these detectors to count 2.5-MeV neutrons requires paraffin shielding to slow the neutrons. The slowing takes place through the scattering of protons by the incident neutrons, a process that typically requires 50 ms to complete. Unfortunately, during this process, called "moderation," all knowledge of a neutron's initial energy is lost. When neutron production occurs in less than 50 ms, the requisite moderation also delays the detection of individual neutrons and masks the time dependence of the production process.

Why then, we might ask, use moderating detectors at all? The answer is that the world is awash in a sea of radioactivity, and many other types of detectors cannot clearly differentiate this radioactivity from neutrons. The other forms of radiation include cosmic rays from stellar objects, including the sun, and gamma rays from radioactivity in the matter that surrounds us. Moderating detectors are supremely efficient at filtering out these other forms of radiation. The tradeoff for such high efficiency is the inability to determine unequivocally what kind of reaction is producing the observed neutrons.

Upon learning of the work at Frascati, researchers at the LLNL Tritium Facility realized that they had the capability to make a decisive impact on the cold fusion controversy. The handling of high-pressure hydrogen gas is part and parcel of the work of scientists and technicians in the facility. High-pressure reaction vessels that could be instrumented safely had already been designed, prototypes had been made, and these vessels could be produced in short order without undue risk. Staff members were experienced in the metallurgical preparation of titanium and palladium; thus, exhaustive tests on the potential of each as a fusion catalyst were feasible. Physicists associated with the neutrino-mass experiment, now under way at LLNL, possessed the expertise to build sensitive neutron analyzers from scratch, and they provided the electronics and software to analyze and integrate data on time scales ranging from a few nanoseconds to half an hour. These capabilities proved to be essential in taking maximum advantage of the thermodynamic monitoring and neutron-detection capabilities of LLNL hardware.

Perhaps our most telling advantage, however, was our ready access to tritium. To appreciate this special advantage, we must understand certain aspects of the two kinds of models theorists had suggested to explain the cold fusion phenomenon. The first model postulated that, by loading a titanium or palladium host to capacity with deuterium, the lattice would force deuterium atoms together. In this model, the fusion rate depends strongly on the masses of the fusing nuclei, which affect the probability that Coulomb repulsion will prevent the reaction. In consequence, the rate of H–D fusion at low temperatures is typically eight orders of magnitude larger than the rate of D–D fusion. The second model assumed that fusion might occur when, for example, temperature-cycling of the lattice caused catastrophic relief of internal stress, a phenomenon referred to as "microfracturing," and proposed that deuterium atoms could forcibly collide in the aftermath of such a fracture. In this hot fusion model, however, we would expect the rate of D–T fusion to be 40 to 170 times higher than the rate of D–D fusion. Enhancements of the D–T reaction due to effects of nuclear structure overcome the mass effects that favor D–D fusion. Repeated measurements of cold fusion with H–D and D–T gas could, therefore, increase experimental sensitivity and provide insight into the mechanisms that might cause fusion. Although many laboratories elsewhere were capable of performing the H–D measurements, the LLNL Tritium Facility is one of the few places in the world that regularly handles high-pressure D–T gas.

Despite our many advantages, executing a quality measurement

was no trivial matter due to the difficulties associated with sample preparation and neutron spectroscopy. In experiments to determine whether uniformity of the host crystalline structure was important, we annealed samples at 800°C for up to one week. To determine whether surface conditioning was important, other samples received different surface preparations. The early results from Frascati and Los Alamos had indicated that active samples might age over time, and most neutron events occurred in the first 20 hours of an experiment. To thoroughly test this observation, we ran our experiments for up to one week.

Compared to the effects seen by Frascati researchers, the neutron bursts reported by later workers were 1000 to 10,000 times lower. This shift in results forced us to make a series of improvements in our neutron-detection equipment. Our initial arrangement (Figure 2) consisted of two identical detectors, each composed of a block of scintillating plastic coupled to a high-sensitivity photomultiplier tube. The scintillator emitted light when exposed to fast neutrons, gamma radiation, and charged particles, such as electrons and muons. The phototube converted the light to an electrical impulse. For most neutrons, the size of the electrical signal is a strong function of the

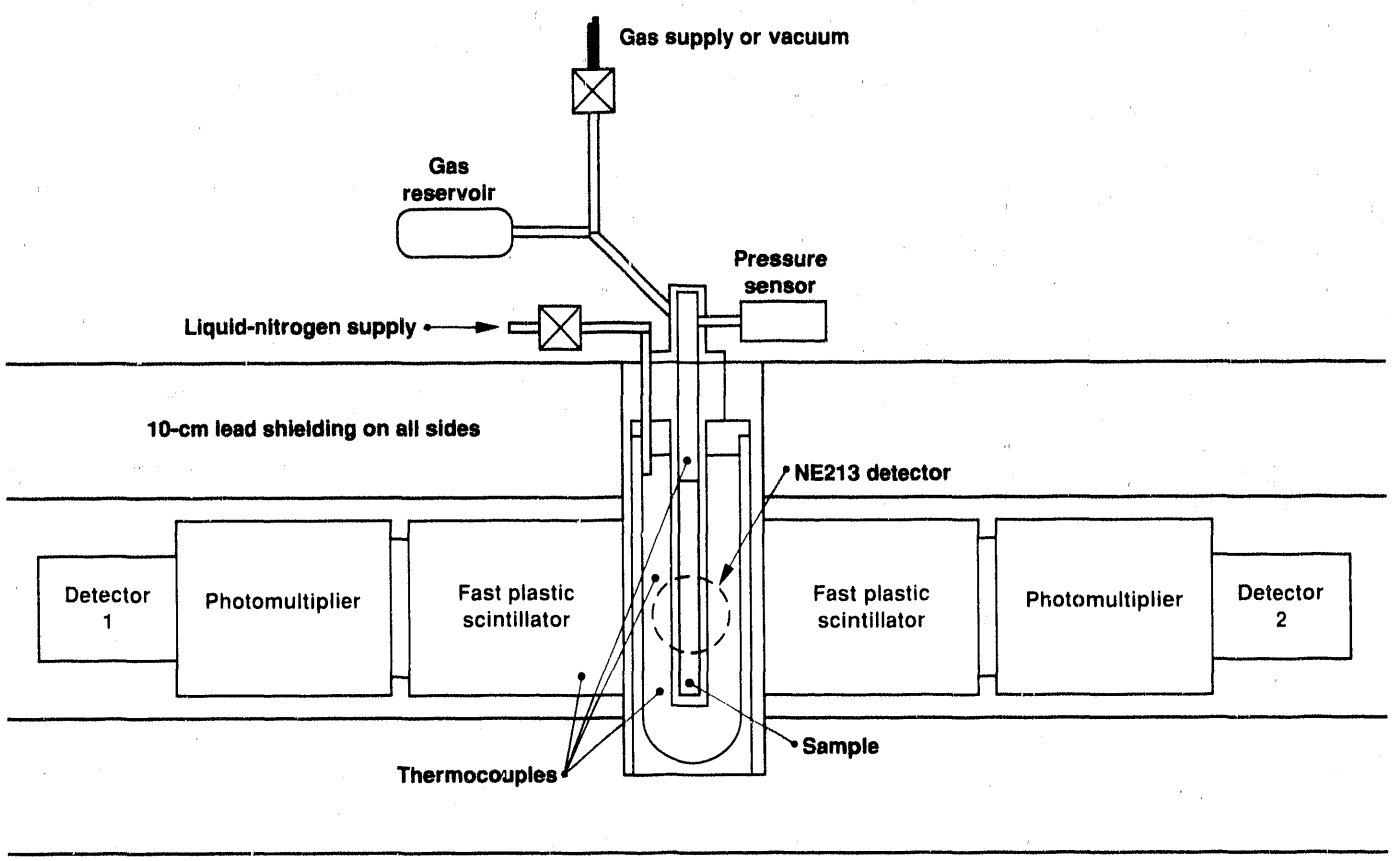


Figure 2. *Experimental setup for measuring neutron flux in deuterium–titanium systems under various conditions of temperature and pressure. Two nuclear-radiation detectors were placed on opposite sides of the cell, which was loaded with hydrogen gas. Each detector consisted of an RCA-8854 photomultiplier tube coupled to a cylinder of Pilot-U fast plastic scintillator, which varied in thickness from 5 to 15 cm in different experiments. In early experiments, we used lead shielding to screen against gamma radiation and did not use neutron-specific scintillants. In the final stages of work, we removed the lead shielding, which was causing a background due to cosmic-ray-induced showers, and we added a neutron-specific detector (the NE213 device). The liquid-nitrogen system cycled the sample temperature between −197°C and room temperature. For some experiments, the cell was pressurized with D_2 gas, and the detectors and electronics were optimized for sensitivity to 2.5-MeV neutrons. Subsequent data were obtained with D–T and H–D gases.*

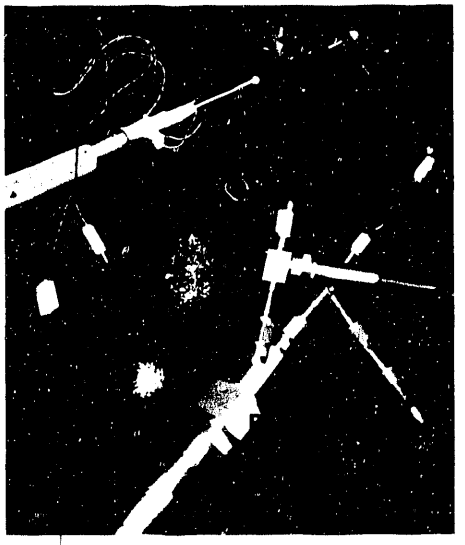
neutron energy. However, the type of plastic we used did not provide good discrimination between neutrons and other forms of radiation. Although other types of plastic could achieve up to 90% rejection of nonneutron signals, we determined that the use of lead shielding to screen against gamma radiation would allow us to detect events nearly 100 times smaller than those seen at Frascati. Thus, we did not employ neutron-specific scintillants at this stage of our work.

Although our detectors (see Figure 3) could resolve particles separated by as little as 30 ns, the electronic components that analyzed the signals could only process one signal every 140 ms. This rate was sufficient to analyze events lasting much longer than 100 ms, such as those seen in the second Frascati experiment, but it was not high enough to analyze short-burst events lasting less than 50 ms. Thus, we adopted a simple particle-counting analysis for these bursts. This scheme allowed us to answer the question of whether there were any instances in which the number of particles detected was anomalously high during any 1-s interval during a given run. For bursts of the type seen at Los Alamos, which were 100 times smaller, we prescreened particles, counting only those that were preceded by two or more particles in the previous 100 ms.

After nearly one month of investigation, we concluded that the lead shielding, which had allowed us to perform sensitive tests for continuous neutron production, also caused spurious signals in the analysis of bursts. In fact, high-energy cosmic rays occasionally interacted violently with the massive lead blocks. These events lit up our detectors with as many as 31 measured particles during a single event. Nevertheless, we were able to measure the energy and time distribution of such particles by recording the detector signals with a digital oscilloscope, thus enabling us to discriminate against those events by comparing their energy signature with that expected for fusion neutrons. Despite the fact that we could determine the nature of the bursts themselves, we were forced to eliminate the lead shielding to obtain maximum sensitivity to the bursts.

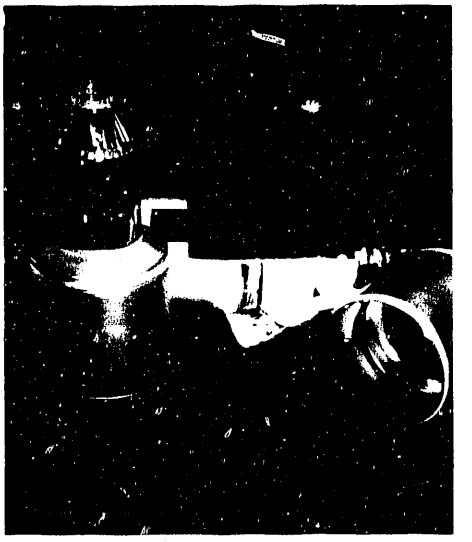
Our work culminated with the use of D–T gas. In these experiments, we incorporated a detector that employed a neutron-specific scintillator, as described earlier, to screen out radiation that was previously blocked by the lead shielding. By using the special

(a) Metal and reaction vessels



• Thermocouples for internal and external temperature monitoring

(b) Neutron detectors



(c) Experimental setup with lead shielding

Figure 3. *Photos of various components in the experimental setup used to investigate possible neutron emission from metal-hydride systems. (a) Metal and reaction vessels used in high-pressure gas cell measurements. The metals shown in the central region, from top to bottom, are palladium wire, titanium lathe turnings, and titanium nuggets. (b) Neutron detectors. This RCA photomultiplier tube and Pilot U scintillator were salvaged from the linear accelerator. (c) Experimental setup, also shown schematically in Figure 2, with lead shielding.*

properties of the scintillator and the knowledge that most particles would not be energetic enough to emulate the signal from a 14.1-MeV D–T fusion neutron, we obtained our most sensitive results for long-lived fusion events. The high energy of the D–T fusion neutron also enabled us to set much more sensitive limits on the size and frequency of burst events.

In summary, we initially found no neutron output during repeated runs using pressurized deuterium cylinders loaded with titanium in various forms. With detector efficiency improved by 15% and using a pair of coincident detectors, we observed a few events that could have been interpreted as bursts of a few hundred fusions. However, the timing of these bursts did not correlate with the temperature of the cylinder, a finding contrary to data from Los Alamos. Further experiments demonstrated that the events were caused by cosmic rays interacting with the lead shielding around our apparatus. Our most sensitive measurements eliminated the possibility of fusion bursts down to levels of 5 fusions per burst. This limit is far below the level reported by Frascati researchers and is also below the much lower rate reported by Los Alamos. Spurious signals often appeared on one detector, but not on another, and such events can be easily misinterpreted as neutrons by inexperienced workers. After 1500 hours of observation, all of our results can be interpreted as consisting of events entirely unrelated to neutron production by a deuterium-titanium system under high gas pressure and thermal stress.

Conclusion

Such an account of our research, however, fails to capture the full flavor of the actual efforts we made. Because cold fusion proved to be something of a moving target, designing the optimal experiment was nearly impossible. Furthermore, our apparatus was assembled under constraints of limited manpower, money, and time. Problems with liquid-nitrogen valves plagued us throughout the experiments, and we were forced to retire several detectors after discovering that they had become entombed in ice overnight when a valve failed to close properly. By the end of our work, we were simultaneously using two kinds of detectors, analyzing them with three different kinds of electronics, and employing six computer programs to control and monitor the equipment and to save data for later analysis. Of the 10 gigabytes of data ultimately committed to tape, we examined nearly 1 gigabyte visually and the remainder was analyzed by a battery of five computer codes. Our work represented an unusual opportunity to demonstrate technical prowess under conditions unconstrained by programmatic requirements. In retrospect, those most intensely involved in this series of investigations may well consider the effort to have been one of the most enjoyable of their professional lives.

Key Words: cold fusion; deuterium-deuterium (D-D) fusion; deuterium-tritium (D-T) fusion; metal-hydride systems.

Reference

1. *LLNL Research on Cold Fusion*, K. I. Thomassen and J. F. Holzrichter, Eds., LLNL Rept. UCID-21809 (1989).

For further information contact Richard Van Konynenburg (above left) (415) 422-0456, Brian K. Balke (above right) (415) 423-5711, or G. Bryant Hudson (left) (415) 423-2947.

Roundtable Discussion on Cold Fusion

On July 9, 1990, several Laboratory scientists who had investigated phenomena purportedly demonstrating cold fusion were invited to discuss their observations. In light of some of the unusual events surrounding research on cold fusion, the E&TR *asked these scientists to comment on a broad range of topics related to the idea of the scientific method as a working mechanism. Their answers reveal a pattern of developments over the past year in which the scientific method itself emerges as the ultimate arbiter. Those present at the roundtable discussion were Bryant Hudson, Brian Balke, Keith Thomassen, and Rich Van Konynenburg. Kent Johnson, scientific editor of the* E&TR, *and two members of the* E&TR *editorial staff were also present. The following is a transcript of their remarks.*

The first claim of success in attaining cold fusion was made to the press by Martin Fleischmann and Stanley Pons at the University of Utah. Their statements were made prior to publication of research findings and before prepublication peer review. What is your reaction to this approach to releasing scientific information?

Keith Thomassen: The original announcement was made on March 23rd, 1989, during a televised press conference that included CNN and all the major network news media. Subsequently, we saw front-page articles in newspapers and reports in magazines, such as *Time* and *Newsweek*. These are not the typical methods for propagating scientific results. For one thing, expectations on the part of the public are raised by that type of approach. It's not something you'd ordinarily do unless you had some pretty sound results, given the major impact that could be expected by an announcement of success in obtaining cold fusion.

Brian Balke: The initial claim also included the statement that they were uncertain about safety and that these were dangerous measurements to make. During the press report, they said, in effect, "We want to prepare people for what might be experienced during these kinds of measurements."

Keith Thomassen: But the impetus was apparently competition between the University of Utah and Brigham Young University. I had the feeling that they thought they had found something quite spectacular and wanted to get a statement out to the press.

Hadn't a manuscript been submitted to a scientific journal before the public announcement, although the peer review hadn't been completed at the time of the press conference?

Rich Van Konynenburg: Fleischmann and Pons did submit a paper to the *Journal of Electroanalytical Chemistry* that was accepted,[1] and what we can presume to be another, possibly longer, paper was submitted to *Nature*. They later withdrew that paper, saying that they didn't have the detailed information requested by the referees. A paper by Jones and his coworkers was accepted by *Nature*.[2]

Keith Thomassen: There was supposed to be a more complete paper from Fleischmann and Pons in the works, but the published paper[1] was quite brief. It circulated almost immediately by FAX, like almost everything following this event. There really wasn't enough scientific content in it to make the case. It was enough to get people started, but it lacked a lot of what would generally be considered good scientific evidence for the claims they made on television.

Brian Balke: I remember seeing a three-page summary analysis of the steps by which you could get to the reported claims of heat production from the initial numbers presented in their paper. It was done by a theorist who was trying to point out that the line of reasoning was not at all straightforward.

Keith Thomassen: Another document that went around early on was their patent application, which had more information on what they were claiming. But like both the television announcement and the original paper, it really didn't substantiate the conclusion about cold fusion.

What was the role of the Department of Energy in attempting to understand the processes and controversy surrounding cold fusion?

Keith Thomassen: Cold fusion became something of a political issue because the national fusion research program receives hundreds of millions of dollars a year, and here a couple of researchers had come along with their own money and had apparently succeeded in making fusion. President Bush was briefed around April 12th or 13th by Glenn Seaborg, a Nobel Laureate and the former head of the Atomic Energy Commission, and the Energy Research Advisory Board panel on cold fusion was asked to recommend to the DOE whether they should put funding into cold fusion. In a letter dated April 24, 1989—one month after the initial announcement—James Watkins, the Secretary of Energy, directed each of the national laboratory directors to use their existing program funds to investigate cold fusion, to give the matter high priority, and to submit weekly reports.

Many researchers soon began reporting other unusual and anomalous effects, which may not have been evaluated properly for alternative explanations. How do you account for this apparent bandwagon effect, for the strong responses from both the scientific community and the public, and for the relatively large number of unusual results?

Keith Thomassen: An intense spotlight was put on cold fusion reports by the prospect of unlimited,

cheap energy from some small gadget you could put together in your kitchen. The probability that they were right was almost zero, but, if they were right, the benefit would be enormous. I think almost everyone recognized that from the outset. So everybody rushed to the lab because it was simple to get an experiment running within a few hours. Like many others, we reviewed the VCR tapes from television, trying to duplicate the setup, and we had a cell running less than 24 hours after we saw the television announcement.

Another feature pervaded the Utah work from the outset. The press release indicated that the researchers were clearly looking for fusion. It wasn't as though they were working on something entirely different and happened to see some new phenomena that could be explained by fusion. It was the other way around—they were trying to produce fusion reactions at the outset. When they saw what they thought was an excess amount of heat coming out of the setup that was too large to be explained by chemical means, they concluded that it must be fusion.

Bryant Hudson: From a sociological perspective, there really hadn't been anything like it before in the history of science, in part, because of today's mass media and things like the FAX. I might add that I got a copy of the Fleischmann and Pons article, shortly after it came out, from my daughter's playschool teacher!

Despite the initial conclusions, there were obvious problems in duplicating the results. What were some of the other problems associated with the early reports?

Keith Thomassen: Even at the time of the initial press conference, the Utah researchers had evidence in hand that their results were suspicious. The suspicious evidence was that the neutron output that should have accompanied a deuterium–deuterium fusion release wasn't there by nine orders of magnitude. Because they persisted in saying it was cold fusion, they had to assume some new type of fusion process that didn't produce neutrons, gammas, or energetic protons.

With respect to the idea of withholding data early on, can you think of any other examples in recent science where important data were withheld because of patent or other considerations?

Keith Thomassen: Not for very long. Even in the high-temperature superconductivity race, where big money was at stake, results were announced quickly.

Brian Balke: In the case of high-temperature superconductivity, the publication was considered proof that they had done original research and was itself a piece of evidence used in the patent process. Once the publication was made, I don't know if anybody really worried about claiming or establishing patents. Another important difference with the high-T_c work is that there were many laboratories doing superconductivity work already, and investigators were well-versed in the problems and difficulties associated with superconductivity measurements. In the case of cold fusion, however, few places had that kind of experience.

Another example that I think is interesting and perhaps a bit more in the line of cold fusion with respect to bizarre or unexpected results is the recent research in Japan measuring the weight of a rapidly spinning gyroscope. The Japanese researchers found that the weight depended on the direction and speed of the gyroscope's rotation, contrary to what our current knowledge of physics predicts. A definitive check on the effect was completed in a few weeks by Jim Fowler at Boulder, Colorado, and his results were negative. He simply designed a gyroscope that spun much faster than the model used in the initial experiment. In that case, you had a macroscopic object and macroscopic equipment, so that it was easy to build and understand what was going on.

However, in the case of palladium hydride, nobody understands in detail what goes on when you squeeze hydrogen into palladium. If you look at the theoretical papers, there was considerable groping around while people were trying to develop a theory that would explain what was being seen, and they couldn't get very far. True, researchers were able to get far enough to demonstrate that the probability for fusion was much lower than what would be necessary to explain the results that Pons and Fleischmann were seeing, but the whole business of the chemistry of metal hydrides can be seen as a black art.

Then one way to account for the positive findings of some and the negative results of many others at the time was the ambiguity concerning the underlying conditioning process of the metal?

Keith Thomassen: In retrospect, as the Energy Research Advisory Board pointed out in their November 1989 summary, *Cold Fusion Research*, enough was understood about the crystallography of palladium to know that the claims of deuterium getting that close together were not valid.

Rich Van Konynenburg: I think one of the things to point out is that there was a time when an individual could claim to have a good grasp of

pretty much all that was known in science. Today, we specialize. One scientist may be a hydride specialist, another a fusion specialist, and somebody else might be an electrochemist, but few people have a complete understanding of all these disciplines. When cold fusion was reported, the tendency was to think about it in terms of what you knew in your own area. Although it didn't look plausible, many thought that there might be something in another area that they didn't know about that was allowing fusion to occur. Since no respected experts stood up and said that the conclusion about fusion was clearly erroneous, giving specific reasons, everybody was willing to give it the benefit of the doubt. The thinking was that maybe the fuzz factor was in some area outside your expertise.

Keith Thomassen: The unusual mix between chemistry and physics is probably one of the more interesting aspects of this research here at the Laboratory. Groups from different disciplines came together, and people started learning very rapidly just the amount they needed to know about somebody else's area that was pertinent to this particular issue. We all gained in the process and eventually became appreciative, as Rich was saying, of the skepticism of experts in all of the other areas.

During the early phases, there seemed to be some friction between chemists and physicists. The physicists tended to be the doubters, and some chemists even reminded the world that it was a chemist who found fission in the first place. What were your observations?

Keith Thomassen: The meetings of the American Physical Society and the American Chemical Society, which took place at about the same time (March 20–24 and April 9–14, 1989), had very different tones. Interestingly enough, Nathan Lewis, a chemist at Cal Tech, was the first one, as I recall, to be bold enough to stand up at a national gathering and reject the idea of cold fusion in no uncertain terms. At the time, most people tended to be rather polite and allowed for the benefit of the doubt, as Rich said. The tendency was not to go with your preconceived ideas or intuition, but to wait until you had done things yourself, had seen the results, and had an opportunity to digest what other people were doing as part of the normal scientific process.

Brian Balke: Not all physicists had a completely negative reaction to the initial claims. When those of us working at the Tritium Facility here at the Lab first heard about cold fusion, the reports seemed so optimistic that, in a sense, we wanted them to be true. Many scientists hope that science can do beneficial things for society. So for a time, we suspended the disbelief that most of us felt. However, I did observe that a number of scientists who had initially seen positive results were eventually frustrated and dismayed by the tremendous publicity they were getting. Some of them clammed up because they regarded their early results as preliminary and didn't feel it was appropriate to subject initial findings to such intense public scrutiny.

Several techniques are available for detecting the byproducts of fusion, such as helium-4, in samples. In fact, the Laboratory was part of a double-blind experiment in which metal samples were analyzed for helium using mass spectrometry. Would you describe this work?

Bryant Hudson: When those of us working in Nuclear Chemistry here at the Lab first heard about cold fusion, we realized that we could play a role in the detection of fusion products due to our work with low-level helium and tritium detection. We felt certain that we could detect the events if they were real, and we knew we had the necessary equipment. We became involved in local experiments and eventually examined samples from a cell run at Texas A&M by Srinivasan. Finally, we were part of a consortium of six mass-spectrometry labs that measured helium in samples of palladium from Fleischmann and Pons at the University of Utah. This final experiment was double-blind in approach, and Pacific Northwest Laboratory (PNL) served as the intermediary.

Although our initial work did not use the double-blind approach, we became confident after the first few studies that we weren't going to see any fusion products. That feeling really does affect how you design the experiment. After all, the difference between our best detection limits and what we should have been seeing from full-fledged fusion was about nine or ten orders of magnitude.

We had a new noble-gas mass spectrometer, the model VG5400 commercial mass spectrometer, and the question was whether or not we would run it with helium-rich samples. We were not told in advance if we would be getting rich samples, but it was reasonable to assume that some of the samples from the University of Utah might contain helium from ion implantation. From our own work on noble-gas ion implantation, we knew it was possible to produce palladium with lots of helium in it. A few days before we got started, we received a phone call from PNL, with whom we were collaborating, urging us to be cautious with the samples because

of the possibility of high helium content. We backed off at that point and went to an older, much less sensitive instrument (the model MS1 commercial mass spectrometer from NUCLIDE Corp.) just to be sure that we didn't ruin a piece of equipment worth a couple hundred thousand dollars. This decision proved to be a wise choice because all the Utah samples had easily measurable helium content.

In the end, the double-blind experiment was not definitive. The palladium wire used in the Utah cells already contained considerable helium as received from the supplier. The final disappointment came when Fleischmann and Pons told PNL two months after all the labs had submitted their helium data that none of the fusion cells in the study had produced excess heat; thus, we shouldn't find any helium. Our paper describing the double-blind experiment has been accepted by *Fusion Technology* and should appear in print soon.

Keith Thomassen: The techniques for discovering whether the products of a fusion reaction are left in a rod are well established, as you point out, and are sensitive by many orders of magnitude. Had the Utah scientists chosen to collaborate immediately, then the controversy might have been settled within a few weeks. It would have determined if there was helium-4 in the rods. Although we offered several times to do the analysis for Pons, he didn't take us up on it. The double-blind experiment on the rods from their laboratory was not begun until many months later. That was discouraging, since it was one of the many ways in which we could have learned more quickly about what had been done if data had been shared from the start.

Bryant Hudson: Lack of communication was a problem in terms of our experiment. By the terms of the double-blind experiment, we weren't allowed to discuss our work with other labs. This hurt the study. But at least now the groundwork is laid so that someone else can do the helium measurements again if they're so inclined. My understanding is that others are following up on our work.

Keith Thomassen: Srinivasan at Texas A&M did one of the first follow-up experiments that indicated there might be some proof of excess heat being produced. We invited Srinivasan here to give a talk on his results, and he presented data that suggested something like 10 or 15% "excess heat" coming out. We then analyzed the palladium rod from that experiment for helium, and the results were negative. This was the first nail in the coffin, from my perspective, and a definitive result.

Brian Balke: Srinivasan also presented some interesting results on sodium deuteroxide. It seemed to me that the evidence he gave showing an effect with lithium deuteroxide, but not sodium deuteroxide, argued for a chemical rather than a nuclear effect. No fusion byproducts were seen.

Keith Thomassen: Srinivasan also replaced the light water with heavy water in the middle of that run. Their calorimetry showed no excess heat until the heavy water was used. When they replaced the heavy water with light water again, the excess heat went away. Although there were many things about that experiment that looked supportive of cold fusion, the helium analysis ruled out fusion.

Rich Van Konynenburg: As far as I know, that result still isn't explained. Srinivasan's experiments indicated that you have to have three things present—a palladium cathode, heavy water in the solution, and lithium. When he substituted any one of those for something that was chemically similar— in one case, just another isotope—then the effect went away.

Keith Thomassen: It's certainly true to this day that there is a residue of uncertainty as to what's going on in fine detail in electrochemical cells. However, I don't think that anyone still claims to be getting more power out of a cell than they are putting in. In their original announcement, Fleischmann and Pons said they were getting about 4.5 times excess heat out over heat in. Then, at the Santa Fe conference (May 23–25, 1989), Robert Huggins from Stanford claimed that he was getting an excess of heat out over heat in.

It's important to realize that most people, in fact, were reporting that heat output was *less* than what was going in, but the output was just a little (10 to 20%) above what they expected to come out. That is, you have to first subtract the power that goes into the electrolysis process itself. This is the power it takes to separate the deuterium and oxygen gases that escape in an open system. After that value is subtracted from the input power, the remainder is what could be called the "true" input, and anything above that is what some people referred to as "excess heat." However, I think that there are too many phenomena going on that aren't under control to make valid claims of excess heat. There were few closed calorimetry systems constructed and operated, and none of those ever showed excess heat.

How does the case of cold fusion research compare with other historical examples of so-called pathological science?

Rich Van Konynenburg: One example that occurred a few years

ago is the case of polywater. The initial report claimed that when water was placed in very fine capillary tubes, it exhibited some unusual properties that could be explained by assuming that it bonded together to form larger molecules. Later, impurities were shown to have caused the observed effects. Although as far as I know, no one at LLNL became involved, we all heard about it. Polywater was something of a curiosity without the tremendous payoff in terms of free energy as implied by cold fusion, so I don't think as many people got involved. Nevertheless, the controversy went on for quite a while before it was settled.

Keith Thomassen: A recent issue of *Physics Today* contained a reprint of a lecture by Irving Langmuir that described a half-dozen attributes of what he called pathological science.[3] One feature is great initial enthusiasm, a large number of people jumping in, and then initial claims disappearing until only a small residue of true believers remains. Another attribute is that, in almost every case, the phenomena in question were at the limit of detection. To me, that is the real essence of the cold fusion argument.

To illustrate this point, the atoms in a molecule of deuterium gas at room temperature are 0.07 nm apart, and the probability of fusion is 10^{-74} events per second per D–D pair. For deuterium in a palladium rod, the number of reactions per second per D–D pair that people were claiming was something like 10^{-24}. That's 50 orders of magnitude higher than the rate for D–D gas at room temperature, while the known separation of deuterium atoms in palladium is more than twice that in the gas molecule. It's curious that the current level of detectability—the best one can do with today's instruments—is exactly where they claimed their output was. One might wonder why nature should be so perverse as to give us 50 orders of magnitude but not, say, 51. At 51, the level would have been ten times above background and everybody would have seen it.

Brian Balke: The level of stability of the instrument is also a factor. I think that few people who have such extremely sensitive detectors ever run them continuously over long periods of time. Most detectors were designed for applications, such as health-physics measurements, in which a fairly high-level signal is present and stability is not a concern. On the other hand, we built an instrument from scratch and didn't make *a priori* assumptions concerning its stability. Each time we saw a signal, we checked and rechecked each connection in our trigger and eliminated many spurious signals by that process. I think that most of the researchers attempted to do good science, but the equipment that we were all working with was barely capable of detecting phenomena at the levels that were being claimed. We at LLNL were able to implement better ways of performing the neutron-detection measurements and point out the importance of having information about the energy of the neutrons being observed.

Do you think recent events will have any long-term effect on the way science and especially publicly funded science will be conducted in the future?

Keith Thomassen: I suspect this matter will largely subside in time, and we will have one more anecdote on how good science should be done and how some science is not done well. Scientists may be reminded to be a little more cautious. If instances of pathological science serve as periodic refreshers of the scientific method, then they probably have some value in that sense.

Thousands of scientific man-hours were spent on attempts to replicate findings and to make better measurements on phenomena associated with cold fusion. In retrospect, was the time well spent or was it wasted?

Brian Balke: That depends on how you interpret what has happened. If the issue is about whether this situation was of great value to the scientific community itself, then I would say that it probably was not. But the value for the public depends on whether people judge cold fusion as a failure of scientists to accurately report data or whether they focus on the fact the science does indeed work. In some respects, the issue is public trust in science. Because some people tend to be suspicious of science, this was a potentially valuable exercise that allowed individuals to see that the scientific process ultimately works.

Keith Thomassen: My reaction is that the scientific response was, in fact, quite proper. This was an issue that raised tremendous expectation in the public sector, and the scientific community responded vigorously and thoroughly. I don't know of a major laboratory in the world that didn't jump in. People spent a lot of time on their own, often after hours, because some of the cells had to be attended on a 24-hour basis. In fact, our experiment started on Good Friday and ran over the Easter weekend. This, despite the fact that, if cold fusion were true, then we would have to reexamine everything we had learned over the last 50 years in nuclear science. Either it was

false, or there was something new and interesting going on. Great discoveries are sometimes made by doing strange things and seeing unusual phenomena, sticking with it, and trying to explain it.

Rich Van Konynenburg: If one tends to become too skeptical of everything, then one doesn't leave open the possibility for discovery. I would also suggest that in ten years or so, another generation of scientists will come along who will never have heard about cold fusion unless one of their professors happened to have been involved and tells his students about it. Such lessons may be forgotten after a generation.

Keith Thomassen: A few interesting technical phenomena were observed, as Brian pointed out. We looked for low neutron rates over extended periods. In our very first cell, we watched the neutron count slowly increase and got quite excited until someone pointed out that the room was getting a little warm. So we turned on a room fan, dropped the temperature a little bit, and the neutrons magically went away. There were many similar situations. Recently, Kevin Wolf, a Texas A&M nuclear chemist, found that his palladium was contaminated with tritium, invalidating his earlier claim of tritium buildup. The Frascati group in Italy was unable to confirm their burst-mode measurements and now say the effect wasn't real.

Brian Balke: Quite often in an experiment, a researcher doesn't understand initially what's going on in an apparatus. If you look through a logbook, you usually find many early false ideas or erroneous assumptions. It's not until you've had some experience that everything becomes clear and you can write down a coherent picture of what was going on.

Do you see science moving in a direction where disagreements are battled out in a court of law when somebody doesn't agree with your results? What role should government play in monitoring science?

Brian Balke: It's not usually necessary to take things to that level. In general, the process of science works because nature is consistent and reproducible. Nature resolves questions of right and wrong, so science can progress without scientists raising questions of personal integrity. Eventually the people within a field learn who does good work and who doesn't.

It is somewhat disturbing, however, to think that the national laboratories and other research institutions could become involved in litigation, because these are the places where much of the very best science is done.

Keith Thomassen: The controversy over who discovered the AIDS virus, HIV, is an interesting example. In that case, President Reagan and French Premier Chirac signed a joint statement saying that the Pasteur Institute and the National Cancer Institute Laboratory under Robert Gallo jointly discovered HIV. At issue were patents held by the U.S. Government for blood tests developed as a result of the research. Similarly, a legal issue might have arisen over cold fusion to establish priority. Had the idea actually been proven, it would've been quite a coup to have CNN carrying your announcement on national television.

With respect to the issue of monitoring science, in most respects, science is a self-correcting process within the scientific community and doesn't generally need outside intervention. I'm not sure that we need intense government scrutiny. The penalties for doing poor science are already severe within the scientific community.

What do you think is the most important lesson learned from this experience?

Brian Balke: The clear implication of the history of cold fusion is that scientists are only human. They seek prestige and rewards for their discoveries. The originators of the cold fusion claims were working in a field that was ripe with potential rewards. Ever since the petroleum crisis of the 1970s, we've had to face the problem of our dependence on fossil fuels. The workers in Utah hoped that they'd found a solution—a cheap, simple, and clean source of fusion power. They went before the world to receive recognition for their accomplishment. Scientists rarely have such an opportunity.

One has to remember that science is a mature institution, and most scientists today are working to find new ways of using the known properties of matter. But—to wax poetic if I may—as the lode of scientific possibility is mined deeper and deeper, the process of science itself changes. Researchers working in one area may break through a barrier only to discover other scientists with a wealth of knowledge in some other field. These encounters are very valuable, and encouraging such cross-disciplinary work should be an important part of managing a modern scientific laboratory.

We need to recognize, however, just how small a part the individual researcher plays in the overall

scientific process. Most scientists are quite cautious, knowing that false or premature claims will be scrutinized by their peers who may have different insights into their work.

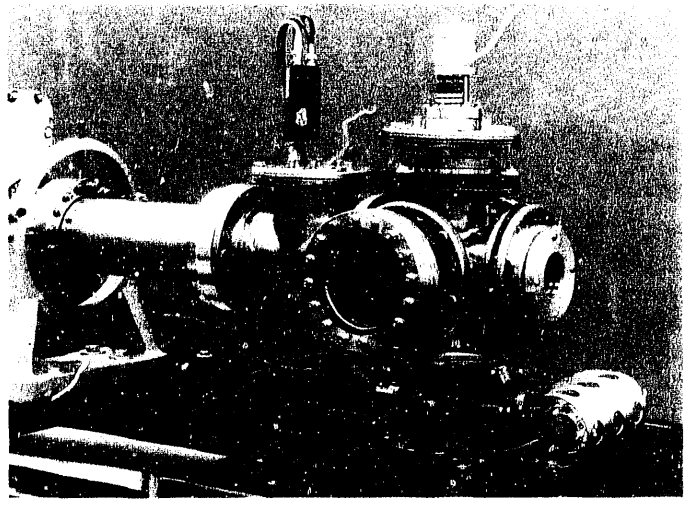
Regardless of what one might wish of the universe, nature is the ultimate judge and jury. Its consistency is mind-boggling. When we attempt to assess the impact of current human behavior, we're talking in terms of only a few decades or maybe a century or two, and very few cultures have ever lasted more than a thousand years. But geological studies of the earth's past reveal a consistency over hundreds of millions of years. And astronomers can observe consistency over billions of years.

Experience tells us that the natural order of our world is fixed, so fitting in with that order is an essential part of a scientist's search for solutions. Whatever the problem, "science" is the best means we have for establishing the nature of the problem and for charting the limits of possible solutions. So when we attempt to evaluate cold fusion, I think we should see not just evidence of human frailty but also the potential for scientists to contribute new solutions to some incredibly challenging problems.

References

1. M. Fleischmann and S. Pons, "Electrochemically Induced Nuclear Fusion of Deuterium," *J. Electroanal. Chem.* **261**, 301–308 (1989).
2. S. E. Jones et al., "Observation of Cold Nuclear Fusion in Conden. ed Matter," *Nature* **338**, 737–740 (1989).
3. I. Langmuir, "Pathological Science," *Phys. Today* **42** (10), 36–48 (1989).

Using MeV Ions To Characterize and Modify Materials



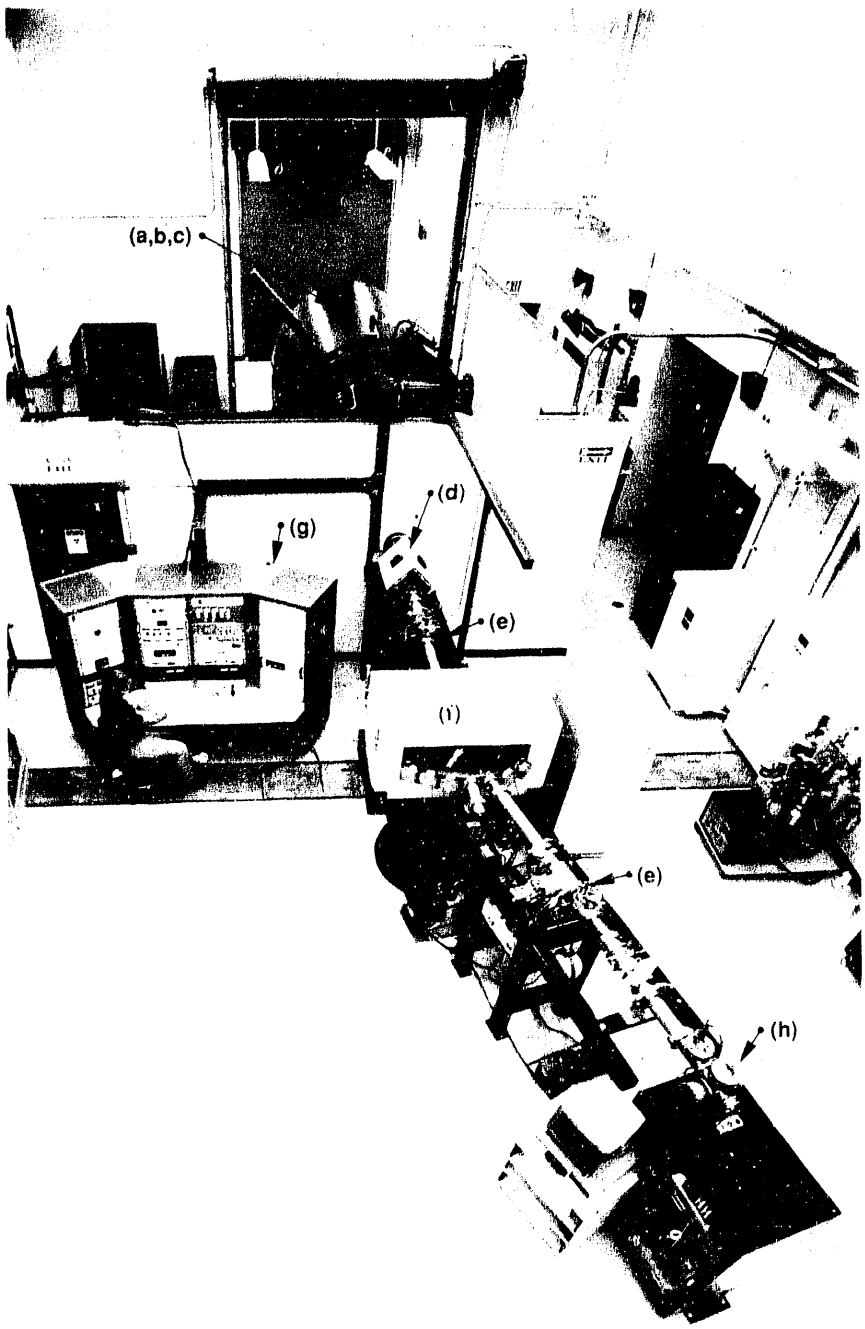
MeV-ion-beam techniques are proving invaluable for characterizing and modifying materials.

Many programs at the Laboratory and elsewhere—for example, nuclear weapon design, space research, and the semiconductor industry—require sophisticated or specialized materials and a precise understanding of the nature of these materials before and after exposure to stressful or exotic conditions. A number of analytical techniques have been developed over the years to probe materials, particularly their layers, interfaces, and internal composition. Some of the most modern techniques make use of ion beams with mega-electron-volt (MeV) energies to characterize materials (i.e., describe them in detail). MeV ions also provide a novel means to modify materials (i.e., alter them to suit special requirements) for fundamental studies and for the development of advanced materials.

The first substantial application of MeV ions in materials research occurred in the semiconductor industry in the early 1970s. The industry needed some way to characterize quantitatively various layers, typically less than one micrometer thick, on silicon substrates without destroying the layers. The probing depth and resolution possible with MeV helium ions using ion backscattering spectroscopy proved to be well suited to this application. Since then, MeV ions have found wide application in such fields of materials science as corrosion and oxidation, coatings, thin films, and surface layers, hydrogen content of materials, friction and wear, material properties and reactions, high-temperature superconductors, semiconductors, and biological and environmental trace analyses.

More than a decade ago, we recognized the value of ion-beam techniques to characterize and modify materials to meet increasingly stringent materials requirements for weapons research and other Laboratory programs. The applicability of MeV ions was demonstrated in the 1980s. These efforts have culminated in the

The New 4-MV Accelerator System



The recently installed 4-MV ion accelerator system gives us the capability to perform all of the ion-beam characterizations and modifications of materials discussed in this article. A photograph of the present system with the main analytical beam line is shown below. This system consists of (a) a 4-MV, single-ended ion accelerator that relies on a pelletron chain for charging, (b) the tank containing the SF_6 insulating gas, (c) a cold-cathode ion source located at the high-voltage terminal potential, (d) a magnetic quadrupole focusing unit, (e) various beam diagnostic and feedback units, (f) an analyzing magnet with chamber to permit selection of the ion to be sent to the specified end station, (g) a control console, and (h) the old, small analytical end station. (In this photograph, Steve Holmes monitors the 4-MV accelerator during an experiment.)

The cold-cathode source can produce ions from any gas. For example, the accelerator can deliver more than 10 μA of analyzed hydrogen, helium, or argon ions and more than 1 μA of doubly charged nitrogen ions to the analytical end station. The present, interim, analytical end station will soon be replaced with a larger, more versatile and automated end station. The analyzing magnet has a mass energy product of at least 500, which means that 3.8-MeV xenon ions can be deflected into the ±15-deg beamlines. The chamber in the analyzing magnet can accommodate up to six analyzed beamlines at ±15, 30, and 45 deg. The other five beamlines will be installed in the future as programmatic and research needs dictate. A beamline for ion-beam modification of materials, including a rastering capability, will be installed on the other 15-deg direction to permit irradiations by energetic, heavy ions.

purchase and installation of a 4-MV ion accelerator system that is dedicated to the characterization and modification of materials (see the box on p. 27). In this article, we first present the relevant basic concepts of ion–material interactions and then summarize the four major techniques used in the ion-beam analysis of materials. We follow by outlining ion-beam modification procedures and discussing examples of some characterizations of materials at LLNL.

Ion–Material Interactions

After monoenergetic ions with energies between 0.4 and 8 MeV enter a material, they begin slowing down by inelastic scattering with electrons (with little rebound) and by elastic scattering with the atomic nuclei (with rebound). The total energy loss by the ions as a function of their depth of penetration into the material (dE/dx) is given by the sum of energy losses for interactions with electrons (dE_e/dx) and with nuclei (dE_n/dx). When the velocity of the ions is much greater than that of the electrons, interactions with electrons dominate and the ion path is considered to be a straight line. For a given ion velocity, dE_e/dx is proportional to the square of the atomic number (Z_1) of the ion. However, as the ion slows, dE_n/dx becomes increasingly important, and, at very low ion velocities, strong deviations from the initial, straight-line flight path occur. The statistical nature of this slowing process leads to a distribution of implanted ions [$N(x)$] with a mean depth, or range ($\bar{R}_p$) and a standard deviation ($\Delta\bar{R}_p$) as shown in Figure 1. For the ion current densities of interest here, the local disturbance (i.e., electronic excitations and atomic motion) caused by one ion ends before the next ion enters the same local zone (typically zones have diameters of 10 to 100 nm).

At incident energies of 0.4 to 8 MeV, essentially all of the ions become implanted. At any depth associated with the straight-line part of the trajectory, the number of ions is preserved; however, their energies decrease slowly and spread increasingly about the average as a result of interactions with electrons. Because the directed ion flux (ions/cm^2/s) can be specified throughout the straight-line path, meaningful material characterizations over this depth can be obtained directly from the known cross sections of rare ion–nuclei interactions and the well-documented values of dE/dx. These values provide a "window" into the material by virtue of the nearly linear relationship between ion energy loss and the amount of material traversed. This relationship forms the basis for depth probing of materials using MeV ions.

Nondestructive MeV-ion-beam techniques are the most direct way to diagnose the surface region (0 to 10 μm) of materials and obtain quantitative information about the distribution or concentrations with depth of the various elements within the material. In contrast to other techniques (for example sputter-

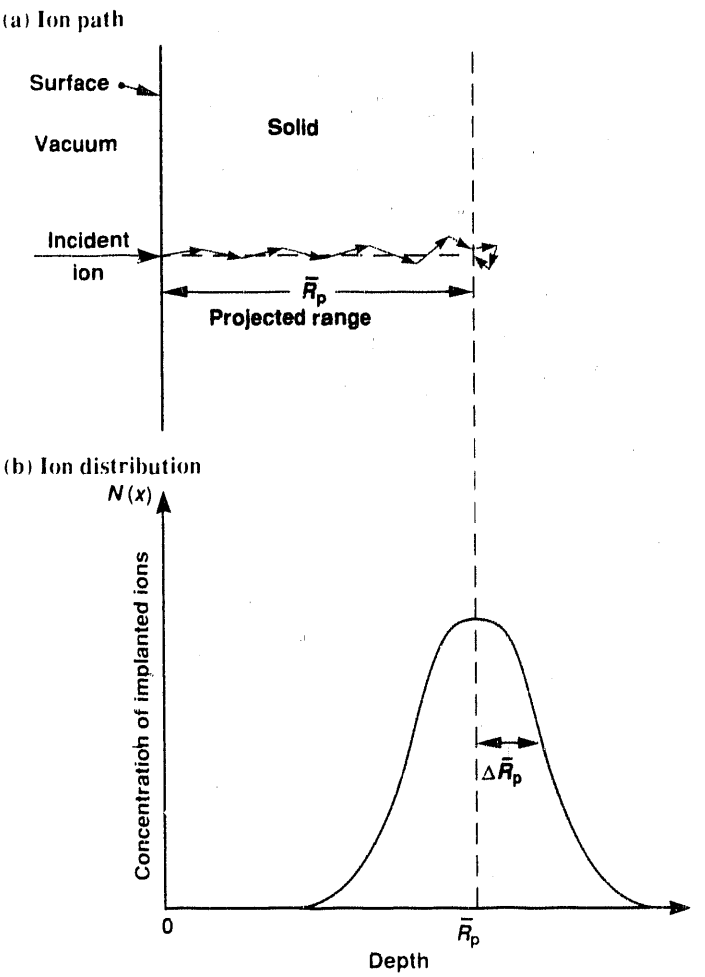


Figure 1. *Typical ion path (a) and distribution of implanted ions (b) in a material.*

profiling techniques such as Auger electron, photoelectron, and secondary ion-mass spectroscopies), the MeV-ion-beam techniques require no specimen preparation and are nondestructive—that is, essentially no material is consumed during analysis. Thus, after a specimen is analyzed, it can be used as planned and can be reanalyzed after any use or treatment. Ion beams also provide a time- and cost-effective way to study a large variety of materials problems and phenomena. These analyses are quantitative because the cross sections for the interactions of the ions with the atoms in the materials are either well known or can be determined using known standards. The depth-distributions of elements are often the key to understanding the processes through which the material was developed or those that have occurred following some treatment or use.

Ion-Beam Analysis

Ions with energies between 0.4 and 8 MeV are well suited to probing materials using four major techniques: ion backscattering (BS), particle-induced x-ray emission (PIXE), ion-induced nuclear reaction analysis (NRA), and forward-recoil spectroscopy (FRS). These techniques are diagrammed in Figure 2. By judiciously selecting the ion species, its incident energy, and the specific ion-beam technique, scientists can tailor the analysis for each element in the periodic table. One or more of these techniques can provide detection sensitivities for various elements of about 10^{-3} monolayer (about 10^{12} atoms/cm^2) for surface layers or less than one atomic part per million (appm) for bulk levels.

All four techniques rely on interactions between the ion and the target atom that occur only when the two particles come within about 0.01 nm—a very small distance compared with the typical interatomic spacing in solids (0.3 nm). A combination of one of these close-encounter analysis techniques together with channeling of ions through the open directions (axial or planar channels) of monocrystalline materials can be used to determine the location of

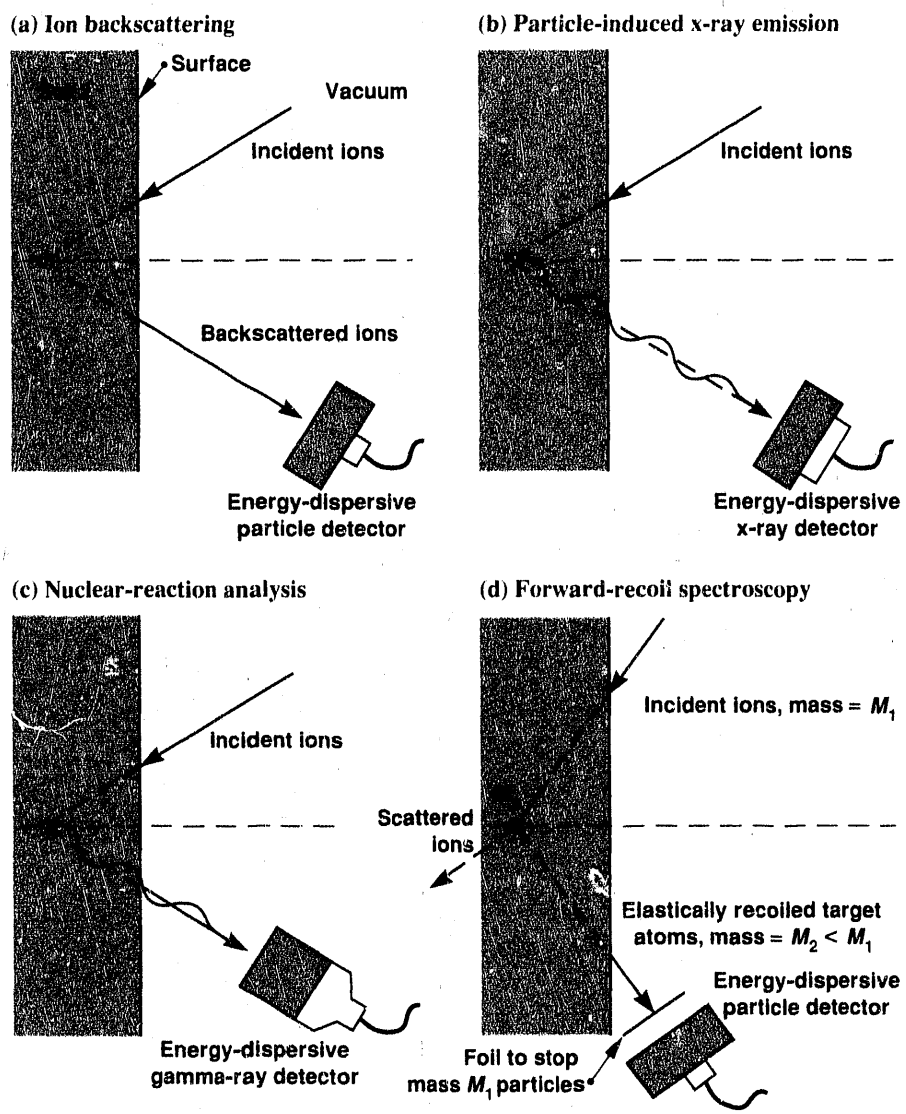


Figure 2. *Diagrams of the four major techniques for characterizing materials using MeV ions as probes.*

impurity atoms in either interstitial or substitutional sites or to assess the extent of lattice imperfections in the near-surface region. This is accomplished by aligning the various crystallographic directions with the ion beam and using one of these four techniques to monitor the signal from the atomic species of interest as the specimen is tilted about the channeling direction. A complete discussion of ion channeling is presented in Reference 1.

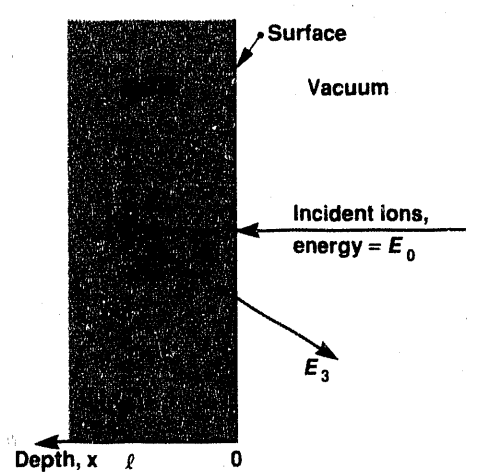
Ion Backscattering

In ion backscattering spectrometry, the energy of ions elastically scattered back from nuclei in the sample is measured. Simple Coulombic or Rutherford backscattering (RBS) using helium ions and protons has proven to be most generally useful. The cross sections for this type of scattering can be calculated using an analytical formula to a precision of much better than 1%. Resonant and nonresonant elastic nuclear scatterings, with interaction cross sections for low-Z (atomic number) target atoms that are more than ten times those for RBS, also result in backscattered ions and can provide unique insights into the composition and properties of some specimens. Interpretation of the backscattering spectra yields information about both the mass and the depth distribution of the elemental constituents of the specimen; resolution is typically about 30 nm for energy-dispersive detectors.

The essence of this energy-loss concept is shown in Figure 3 for the case of an ion with an energy E_0 incident normal to the surface of a homogeneous material, backscattered from an atom at a depth ℓ, and detected with an energy E_3. Just before scattering at depth ℓ, the ion has an energy

$$E_1 = E_0 - \ell\, dE_{in}/dx \ ,$$

where, for shallow depths, the value of dE_{in}/dx is assumed to be that for E_0 because dE/dx is typically a slowly varying function of energy.



$$E_1 \simeq E_0 - \ell \left.\frac{dE}{dx}\right|_{E_0}$$

$$E_2 = kE_1$$

$$E_3 \simeq E_2 - \frac{\ell}{\cos\theta} \left.\frac{dE}{dx}\right|_{E_2}$$

$$\therefore E_3 \simeq kE_0 - k\ell \left.\frac{dE}{dx}\right|_{E_0} - \frac{\ell}{\cos\theta} \left.\frac{dE}{dx}\right|_{E_2}$$

Figure 3. *Relationship between ion energy loss and depth of scattering event. The equations are described in the text.*

Immediately after scattering, the ion has an energy $E_2 = k\,E_1$, where the kinematic factor k is the fraction of energy retained after scattering through the indicated angle; k can be calculated using the ion and target-atom masses in relations conserving energy and momentum. Thus,

$$E_3 = E_2 - \ell/\cos\theta\ dE_{out}/dx \ ,$$

where dE_{out}/dx can be evaluated at E_2. Combining these relations, the energy E_3 of the detected ion is linearly related to the depth ℓ by

$$E_3 = k\,E_0 - k\,\ell\ dE_{in}/dx - \ell/\cos\theta\ dE_{out}/dx \ .$$

A detailed discussion of ion backscattering spectrometry for materials analysis is given in Reference 2.

Particle-Induced X-Ray Emission

Particle-induced x-ray emission (PIXE) is characteristic of the atomic species in the bombarded target and results from inner-shell ionizations. The bremsstrahlung background in the x-ray spectrum produced by ion excitation is greatly reduced compared to that produced by electron excitation. Consequently, the detection limits using PIXE are ten to ten thousand times better than those using electron excitation.

PIXE and RBS are well matched as complementary techniques because, for a given ion energy, the x-ray production cross section (σ_X) decreases dramatically with the atomic number Z of the target atoms (e.g., $\sigma_X \sim 1/Z^8$ for K-shell x-ray production), whereas the Rutherford cross section (σ_R) increases as the square of the atomic number (Z^2). In addition, PIXE provides a clear distinction between atoms with similar atomic numbers, while

RBS does not always yield an unambiguous identification for high-Z atoms.

The PIXE signal represents the integral of all the x rays created along the ion's path, corrected for photoelectric absorption of the x rays by the material. Thus, PIXE only permits depth profiling with very poor resolution (about 100 nm). In principle, PIXE could be used for the analysis of all elements with $Z > 2$; however, the use of energy-dispersive, lithium-drifted, silicon [Si(Li)] detectors limits the range to those elements with $Z > 5$. Because both x rays and backscattered particles emanate from the specimen surface during ion irradiation, both should be detected. In fact, we have clearly demonstrated the compatibility and usefulness of simultaneous x-ray and particle spectroscopies.[3,4]

Ion-Induced Nuclear-Reaction Analysis

Ion-induced nuclear reactions often result in the prompt emission of reaction products (such as ions and/or gamma rays) that are uniquely related to the nuclei of the reacting particles (the incident ions and the target atoms). Thus, nuclear-reaction analysis (NRA) consists of energy spectrometry of the reaction products and yields an unambiguous identification of the reacting nuclei. Because the reactions are isotope-specific, the background signals tend to be very low and the sensitivity for the desired isotope can be quite good, even though the cross sections are typically orders of magnitude smaller than those for RBS or PIXE. In contrast to RBS, there is no simple analytical expression for the cross sections, and standards must be used to analyze the materials. When the relative velocity of the ion and target atom are sufficiently high, the Coulomb barriers of the two nuclei may be interpenetrated and an ion-induced nuclear reaction may occur. For ion energies of less than 8 MeV, NRA has been applied to elements with Z between 1 and 15.

The reaction cross sections of many ion-induced nuclear reactions have sharp resonances as a function of ion energy. If these resonances have a narrow energy width and are well separated in energy, then depth profiling can be performed by starting with the ions whose resonance reaction occurs at the surface. Then, as the energy of the ion is increased, the resonance can be placed at increasingly greater depths in the sample. Because the reaction yield at each depth is proportional to the concentration of the isotope of interest, the isotopic depth distribution can be determined from the known energy loss of the ion in the material. Quantitative, nondestructive depth profiling of ordinary hydrogen with high depth resolution (~3 nm) using nitrogen-15 ions, which have a resonance at 6.38 MeV, illustrates how NRA complements RBS and PIXE for analyzing low-Z elements in high-Z materials.

Forward-Recoil Spectroscopy

When the mass of the incident particle is equal to or greater than the mass of the target atom, elastic backscattering cannot occur. However, a large part of the incident ion energy can be transferred to the lighter target atom, which then recoils into a forward angle. In forward-recoil spectrometry (FRS), energy spectroscopy of these elastically recoiled atoms yields the initial depth distributions of the recoiled atoms. Generally, the incident ion and detected recoiled atom have paths at small angles (<30 deg) with the surface of thick specimens. Thin, freestanding foils are an exception, because the recoiled atoms can escape through the foil to a detector. FRS allows all elements to be analyzed with a depth resolution of about 100 nm and much higher cross sections than those for NRA. Thus, FRS is very useful for surface and bulk analysis of isotopes of hydrogen and other light elements when depth resolution is not critical.

Ion-Beam Modification

High-energy ion-beam modification of materials can be divided into two distinctive classes: implantation and irradiation. Although essentially all of the ions become implanted in both classes, implantation includes procedures for which the implanted layer is the desired end effect. Examples of implantation applications are calibration standards for analysis techniques and other experiments, low-concentration doping, synthesis of compounds or alloys, and formation of subsurface elemental layers. Typically, the fluences (ions/cm^2) used in these implantation procedures vary from about 10^{13} to greater than 10^{18} ions/cm^2.

Irradiation includes processes in which the important features are the interactions of the ions with the material before they come to rest. Two examples are radiation damage and mixing and/or stitching at interfaces. Ions create radiation damage by displacing atoms from their equilibrium positions; the resulting defects affect both the mechanical and electrical properties of materials. Passage of ions through interfaces between dissimilar materials can lead to the formation

of interfacial alloys or compounds as a result of atomic intermixing caused by collision cascades and radiation-enhanced diffusion. In addition, mixing and stitching can be used to enhance the adherence of a coating to a substrate as ions are passed through the interface. The term "mixing" is generally used to describe this enhancement phenomenon when the dominant energy deposition process at the interface leads to atomic displacements, because significant atomic exchange across the interface is expected. When electronic excitation dominates, very little atomic exchange occurs between the coating and the substrate, but the electronic bonding configurations across the interface are modified. For all of these irradiation cases, the fluences rarely exceed 10^{16} ions/cm^2.

In practice, uniform modifications are achieved by rastering the ion beam over the region of interest, which may have a diameter between about 1 and 20 cm. Historically, most modifications have been performed using 20- to 200-keV ions, because the lower energy machines can deliver higher currents. Our new accelerator (0.4 to 4 MV) cannot produce the higher currents but it can provide reasonable currents for all but the highest fluence applications. However, the higher energies mean greater ion ranges, which may be important for some applications. Although we have used our 200-kV implanter for a variety of modification studies, we have not yet used MeV ions for any ion-beam modifications of materials. Consequently, in this article, we

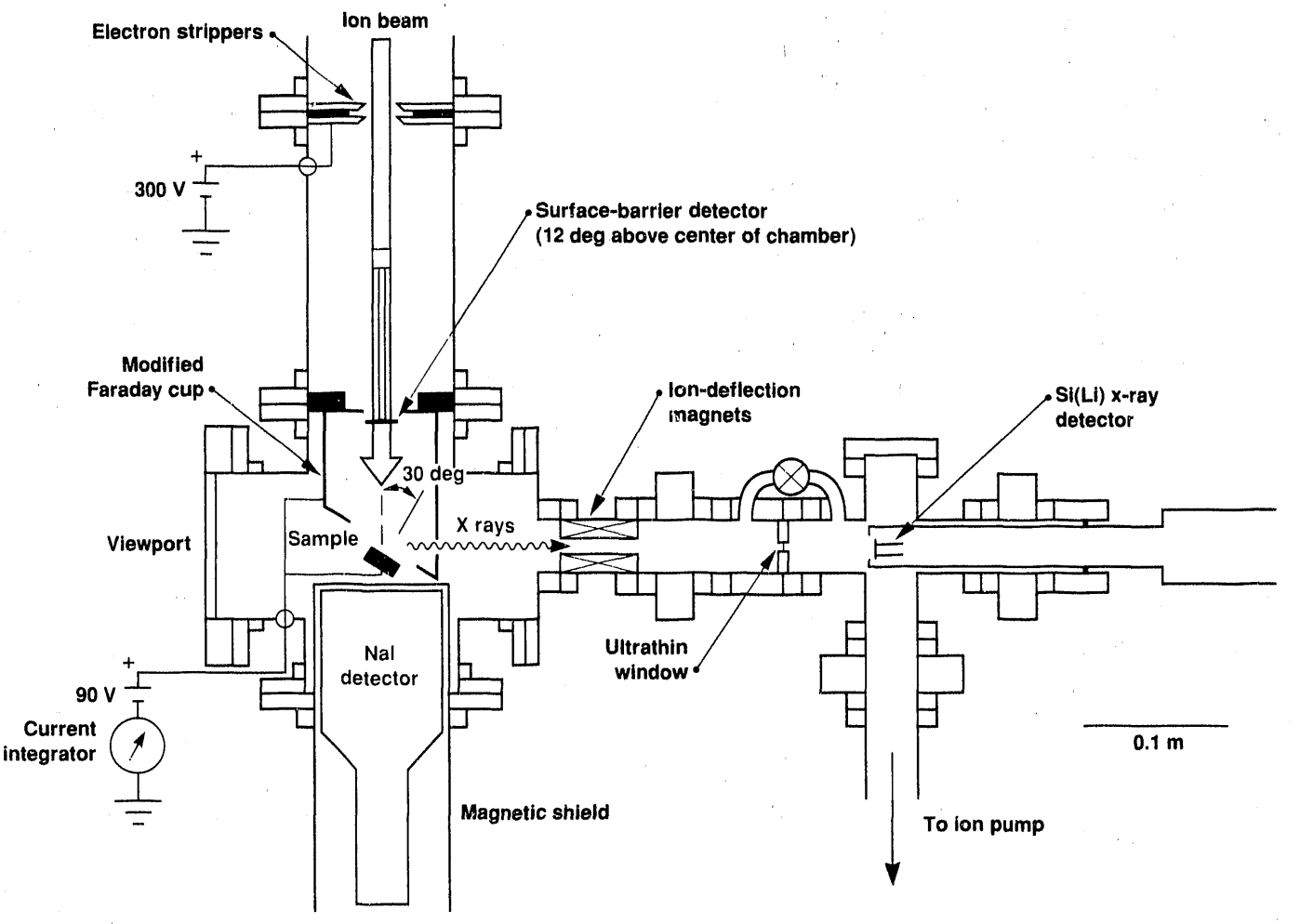


Figure 4. *Top view of experimental arrangement for ion backscattering, particle-induced x-ray emission, and nuclear-reaction analysis.*

emphasize materials characterization in which MeV ions are used as probes.

Ion-Beam Materials Characterization

MeV-ion-beam techniques are being used to provide important materials information, including:
- Quantitative, nondestructive depth profiles of the elements in materials.
- Reaction rates for gases with materials and for solid materials in contact with each other by analyzing the solid reaction products.
- Measures of crystalline perfection.
- Diffusion constants and activation energies.
- Solubilities.
- Lattice locations of atoms.
- Pore sizes in microporous materials.

During the 1980s, we used MeV ions from a 3-MV accelerator, which no longer exists, to probe materials and obtain for a variety of LLNL projects one or more of the first three types of information listed above. The applications included analysis of foils and coatings for x-ray scattering experiments and other purposes, coatings and interfaces related to bonding and adhesion failures, polishing damage on the surface of crystals, ion-, electron-, and laser-modified materials, and materials after they had reacted with gases. Representative examples of these analyses are discussed below.

A schematic of the experimental arrangement used to obtain most of these results is shown in Figure 4. Typically, the specimen surface is at an angle of 60 to 75 deg from the ion-beam axis and is tilted toward the axis of the ultrathin-windowed Si(Li) x-ray detector. The energy-dispersive, surface-barrier detector for the particles backscattered through 168 deg is located directly above the ion-beam axis. Gamma rays resulting from nuclear reactions are detected by the NaI detector after they penetrate through the chamber wall. A modified Faraday-cup arrangement (with the indicated biasing) permits accurate dosimetry during the measurements.

Carbon Foil Composition and Thickness

We determined the composition and thickness of a free-standing, deposited carbon foil by simultaneous RBS and PIXE using 2-MeV helium ions.[5] In the backscattering spectrum shown in Figure 5, the subscripts "T" and "B" refer to the energy position for helium scattered from the indicated element at the top or base of the foil, as originally deposited on a substrate. The corresponding PIXE spectrum revealed peaks for C(K), O(K), Si(K), Ar(K), Fe(L), and Cu(L) x rays. This knowledge of what elements are present removed uncertainties related to the interpretation of the RBS results, which show that contamination by oxygen (1.6 at.%), argon (0.2 at.%), iron (0.5 at.%) and copper (0.6 at.%) exists uniformly throughout the entire foil thickness. In contrast, there is a very small backscatter peak caused by silicon inside the foil near the center. We made a quantitative evaluation of the carbon-atom areal density (4.62×10^{18} carbon atoms/cm^2 or 92.0 μg carbon/cm^2) and the amounts of the impurities, assuming that the stopping matrix is pure carbon. Taking 2.0 g/cm^3 as the density of the deposited carbon converts the areal density obtained by RBS to a thickness of 460 nm. Information gained from such measurements on both single-layer and multilayer foils is essential if we are to optimize foil fabrication and

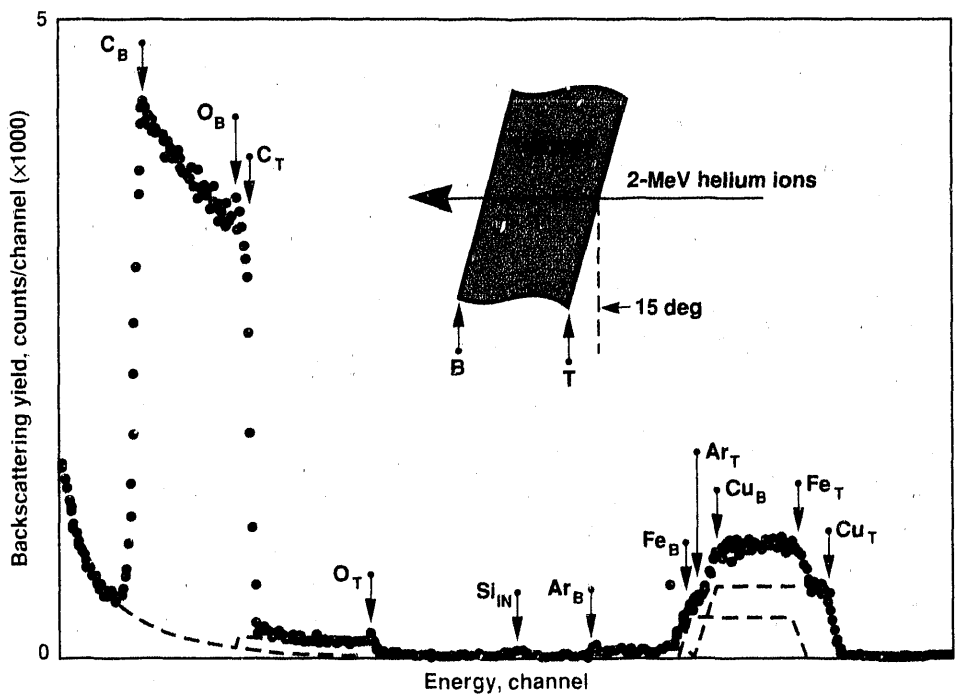


Figure 5. *Helium-ion backscattering spectrum for free-standing, 460-nm-thick carbon foil.*

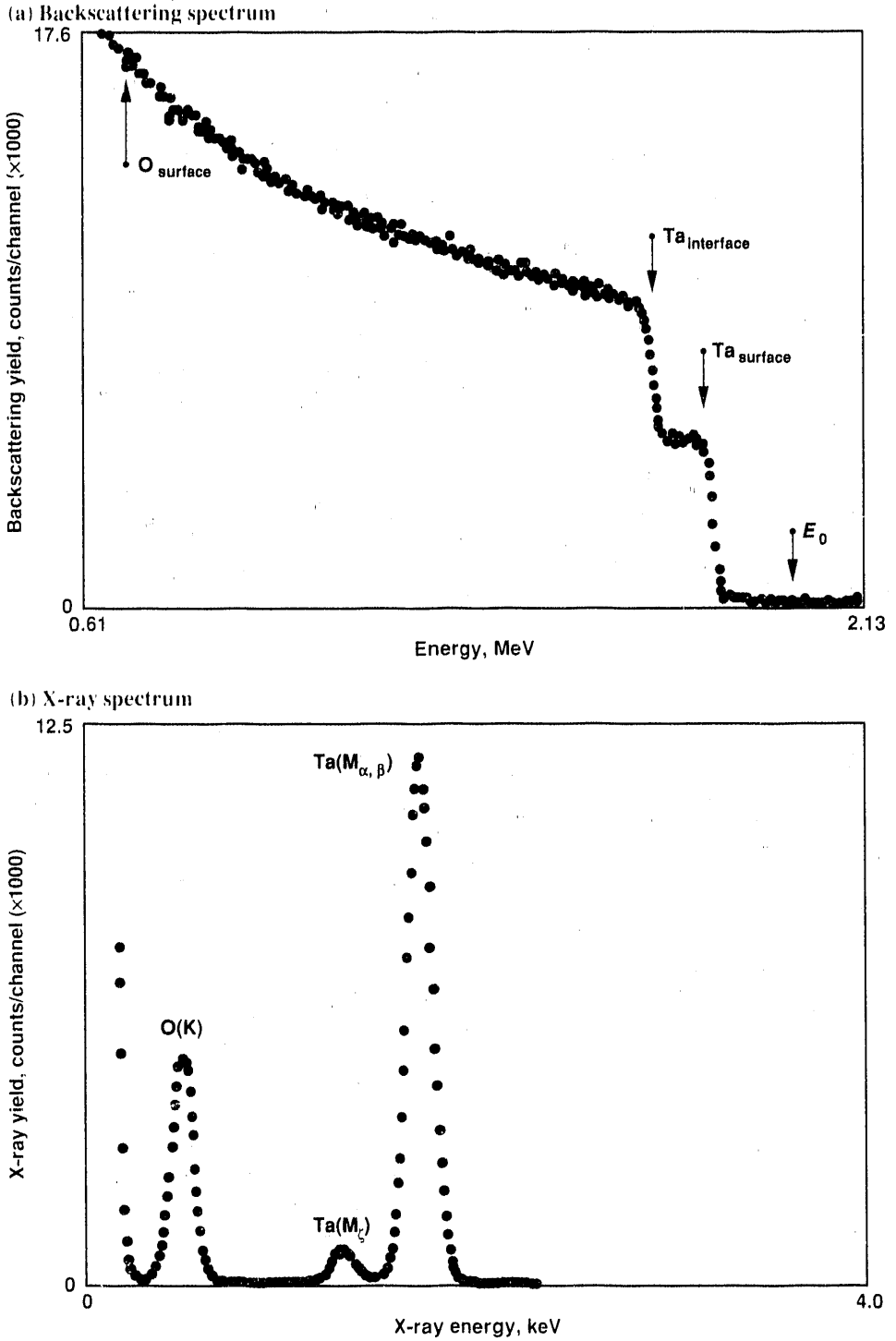


Figure 6. *Simultaneous RBS and PIXE analyses of anodized tantalum for a fluence of 1.5×10^{15} helium ions/cm² over a 2-mm-diameter spot: (a) backscattering spectrum, and (b) x-ray spectrum. The analyses indicated a Ta_2O_5 layer 119 nm thick and an oxygen content of 18.7 μg oxygen/cm².*

storage procedures and ensure the correct interpretation of the results from x-ray scattering experiments.

Simultaneous RBS and PIXE of Surface Layers

The value of using simultaneous RBS and PIXE to eliminate ambiguities in RBS results from high-Z materials with surface layers containing low-Z elements can be demonstrated by analyzing anodized, high-purity tantalum.[3] The results of such analyses are given in Figure 6. The absence of any definitive indication of the presence of oxygen in the backscattering spectrum (Figure 6a) is simply a consequence of the small relative cross section for scattering from oxygen (cross section approximately 1% that for tantalum) and the statistics of the data (about 1%) at the energy corresponding to scattering from oxygen at the surface.

Because only two edges and one plateau are apparent in the RBS spectrum, the spectrum could represent either: (1) a uniform surface compound of a high-Z element (tantalum in this case) and an unknown low-Z element on a substrate of the high-Z element with the edge labeled $Ta_{interface}$ corresponding to scattering from the high-Z atoms at the interface between the compound and the base material, or (2) a uniform alloy of the high-Z element with a lower-Z element with the edge labeled $Ta_{interface}$ corresponding to scattering from the lower-Z atoms (rhodium) located at the surface. Even the identity of the high-Z element is uncertain because the kinematic factor for scattering from high-mass (high-Z) atoms varies slowly with mass. For the given experimental conditions, surface scattering from hafnium or tungsten would give an edge within one channel of that shown for tantalum.

The x-ray results in Figure 6b show that the surface region of the sample contains only tantalum and oxygen and that the RBS results are, in fact, indicative of a surface compound of tantalum and oxygen with a uniform, in-depth composition. Although only the x-ray spectrum for the 0- to 4-keV region is shown in this figure, we took data for 0 to 10 keV; all observed peaks were characteristic of either oxygen (K x rays) or tantalum (L and M x rays). With the ambiguities removed, we applied standard RBS data reduction procedures[2] to the results to determine that the surface layer contains 18.7 μg oxygen/cm^2, which corresponds to a Ta_2O_5 oxide thickness of about 119 nm.

Oxides of Uranium Exposed to Water Vapor

In a study of the oxidation of uranium in water vapor, 2-MeV helium-ion backscattering gave useful results for UO_2 thicknesses between about 30 and 1000 nm.[6] For thicker oxides (up to about 5 μm), 2.5-MeV proton backscattering provided the necessary probing depth. For oxides thinner than 30 nm, we used PIXE to quantify the oxygen surface density by comparing it with thin oxide standards on silicon. Figure 7 shows that an exposure to ambient air of less than 30 min after electropolishing results in an oxide equivalent to 20 nm of UO_2, which increases slowly with further exposure to ambient air. Just 1 h at 80°C in 13 kPa of water vapor increases the oxide thickness to 106 nm. The 3σ detection limit for oxygen by PIXE is 0.08 μg oxygen/cm^2, which corresponds to 0.6 nm of UO_2. Independent backscattering and PIXE results generally agreed within 15% for UO_2 thicknesses between 30 and 100 nm.

Oxygen Contamination on and in Beryllium

Low levels of oxygen on and in beryllium foils can affect the results of certain x-ray scattering experiments. Surface and bulk levels of less than 10^{15} oxygen atoms/cm^2 and 50 appm, respectively, were considered significant. Thus, the measurement goals were to determine surface oxygen with a detection limit of a few times 10^{14} oxygen atoms/cm^2 and bulk oxygen with a sensitivity approaching 10 appm in a volume large enough to be representative of the bulk material (e.g., 5 mm × 5 mm × 2 μm deep). Oxygen on beryllium at the indicated level can be analyzed routinely using helium-ion backscattering; however, using this technique, we can determine the bulk oxygen concentration only at levels above about 350 appm.

We performed simultaneous RBS and PIXE measurements using 2-MeV helium ions to improve the detection limit for bulk oxygen.[4] RBS measurements allowed us to determine the surface oxygen before and after *in situ* sputter cleaning by 3-keV argon ions in an ultrahigh-vacuum system. PIXE measurements of specimens with surfaces maintained clean by sputtering allowed us to assess the concentration of oxygen in the bulk. Because the RBS is quantitative and the PIXE results can be directly related to an analytical calculation without any unknown parameters, this combined approach is quantitative. We eliminated unknown PIXE parameters by relating the change in the surface oxygen level (as determined by the RBS) following sputter cleaning to the corresponding change in the oxygen x-ray intensity.

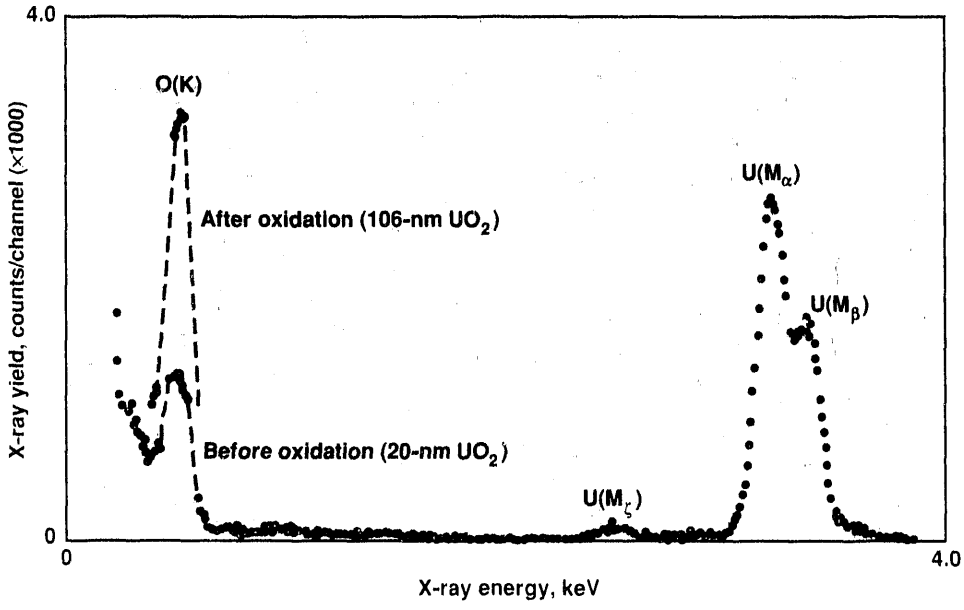


Figure 7. *X-ray spectra generated by 2-MeV helium ions for electropolished uranium before (a) and after (b) oxidation (1 h at 80°C in 13 kPa of H_2O). The thickness of the UO_2 layer increased from 20 nm to 106 nm.*

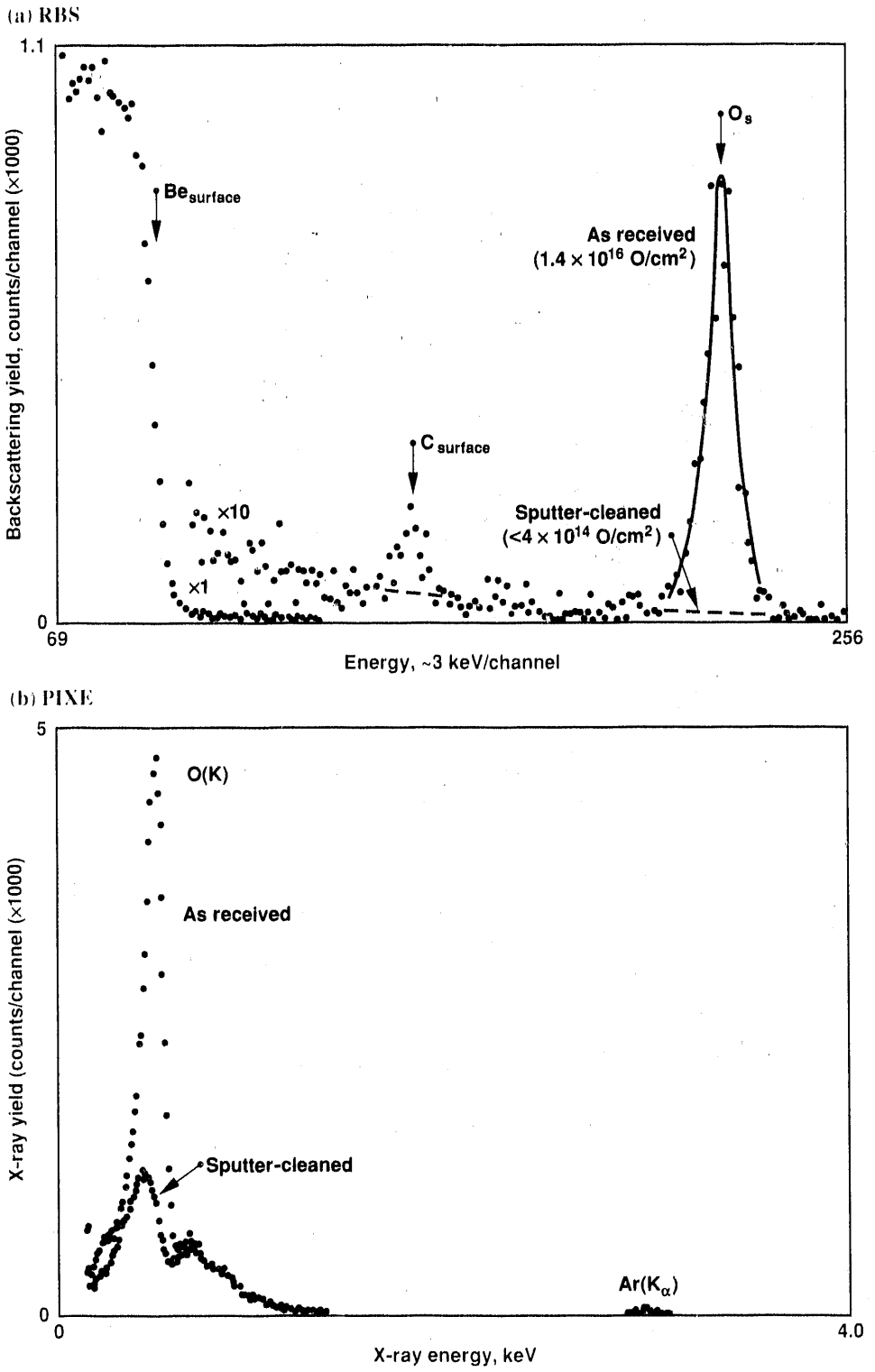


Figure 8. *Simultaneous (a) RBS and (b) PIXE of a beryllium foil in the as-received (chemically etched) and sputter-cleaned conditions. The as-received beryllium had a BeO oxide layer with a thickness of 19 nm, and the bulk oxygen level was 220 appm.*

Figure 8 shows the RBS and PIXE results, which indicate that the beryllium foil had 1.4×10^{16} oxygen atoms/cm^2 on the surface (equivalent to 1.9 nm of BeO) and 220 appm oxygen in the bulk material. The 3σ bulk detection limit using PIXE is 10 appm. This low detection limit is a direct consequence of the low x-ray background in the PIXE spectra and the removal of the surface oxygen by sputtering, which was critical because the oxygen x-ray signal from 1.9 nm of surface BeO is equivalent to that from 600 appm of oxygen in the bulk material.

Profiles of Aluminum Implanted in Tantalum

In a study of the effects of ion implantation on the oxidation of tantalum,[7] we profiled aluminum in tantalum using the nuclear-resonance reaction that yields gamma rays when 0.998-MeV protons react with aluminum atoms. Figure 9 shows the concentration profiles for aluminum in tantalum following implantation and after oxidation. We determined the concentration scale using a pure aluminum target as a standard. These results indicate that, during high-temperature oxidation, the implanted aluminum diffuses rapidly away from the near-surface region of the tantalum. The results also explain why the implanted aluminum does not measurably affect the oxidation of tantalum under these conditions.

Summary

The Laboratory has a significant capability for characterizing and modifying materials using MeV ions beams. A new 4-MV accelerator system dedicated to this work has been installed recently; it joins a 200-kV ion implanter. The quantitative, nondestructive nature of the major ion-beam analysis

techniques is proving invaluable for resolving a variety of materials problem and for developing new materials.

Key Words: ions—4-MV accelerator system, megaelectron volt (MeV); materials characterization; materials modification; MeV-ion-beam techniques—forward-recoil spectroscopy, ion backscattering, ion-induced nuclear-reaction analysis, particle-induced x-ray emission.

References

1. L. C. Feldman, J. W. Mayer, and S. T. Picraux, *Materials Analysis by Ion Channeling* (Academic Press, New York, 1982).
2. W.-K. Chu, J. W. Mayer, and M. A. Nicolet, *Backscattering Spectrometry* (Academic Press, New York, 1978).
3. R. G. Musket, "Applications of Simultaneous Ion Backscattering and Ion-Induced X-Ray Emission," *Nucl. Instr. Meth.* **218**, 420 (1983).
4. R. G. Musket, "Analysis of Oxygen on and in Beryllium Using 2-MeV Helium Ions," *Nucl. Instr. Meth.* **B24/25**, 698 (1987).
5. R. G. Musket, "Composition and Thickness of Multilayer Foils Using MeV Ion-Beam Techniques," *Proc. SPIE* **911**, 177 (1988).
6. E. N. Kaufmann, R. G. Musket, C. A. Colmenares, and B. R. Appleton, "Enhancement of Uranium Oxidation Resistance by Molybdenum Implantation," *Mat. Res. Soc. Symp. Proc.* **27**, 747 (1984).
7. E. N. Kaufmann, R. G. Musket, J. J. Truhan, K. S. Grabowski, C. R. Gossett, and I. L. Singer, "High-Temperature Oxidation of Ion Implanted Tantalum," *Nucl. Instr. Meth.* **209/210**, 953 (1983).

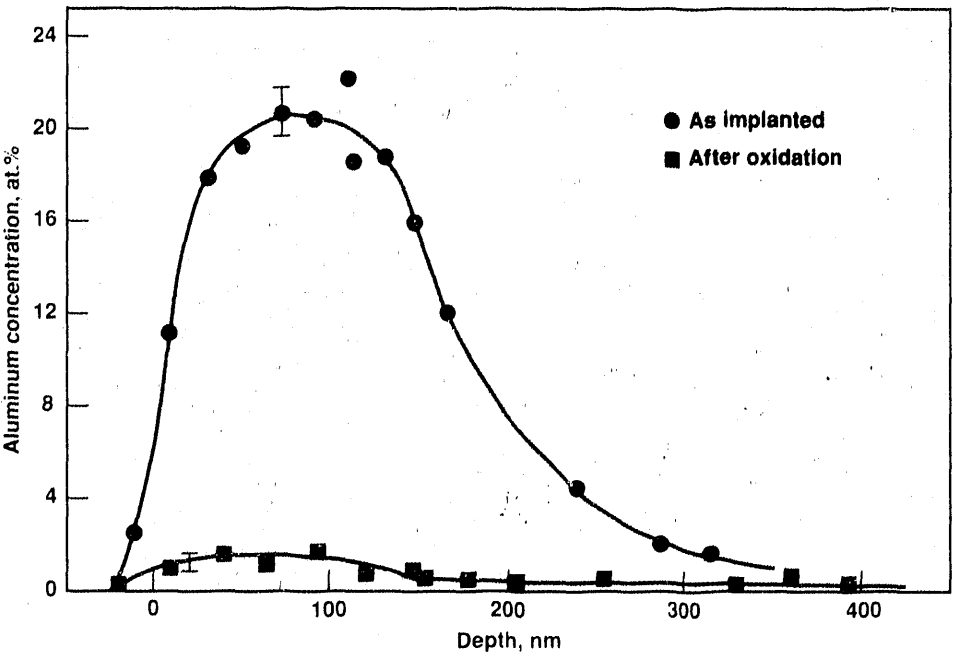


Figure 9. *Depth profiles of aluminum in tantalum using the (p,γ) nuclear-resonance reaction. The implanted fluence was 2 × 10^{17} aluminum atoms/cm² at 150 keV. The circles show the aluminum concentration as implanted; the squares show the aluminum concentration after oxidation at 1000°C for 5 min in 100 kPa of flowing oxygen gas.*

For further information contact Ronald G. Musket (415) 422-0483.

Abstracts

History of Cold Fusion Experiments

On March 24, 1989, two scientists from the University of Utah announced the achievement of "cold" fusion, a new nuclear fusion process made possible using an inexpensive device and conventional materials. The implication of their claim—unlimited, cheap energy—motivated scientists around the world to rush to their laboratories and attempt to reproduce the Utah results. During the months following, when cold fusion activity was at its peak, there were many instances when encouraging preliminary results were announced and retracted shortly thereafter. Retractions were usually the result of finding errors in the interpretation of data. Even though the general concensus now is that the phenomenon of cold fusion was a false hope, a number of redeeming aspects emerged from the search, including a better understanding of instrumentation errors, the difficulties of calorimetry, and the complexity of palladium hydrides. Few other efforts in recent scientific history have rivaled the level of activity generated by the cold fusion controversy.

Contact: Keith I. Thomassen (415) 422-9815.

LLNL Experiments on Cold Fusion

A series of experiments and calculations were made at LLNL shortly after the March 1989 announcement of cold fusion by researchers at the University of Utah and Brigham Young University. We found no evidence for significant rates of nuclear fusion reactions near room temperature or under conditions of high pressure and temperature cycling. Instead, we found that a variety of observations can be traced to such experimental artifacts as temperature sensitivity of neutron detectors and catalytic oxidation and heating of palladium cathodes.

Contact: Richard Van Konynenburg (415) 422-0456, Brian K. Balke (415) 423-5711, or G. Bryant Hudson (415) 423-2947.

Roundtable Discussion on Cold Fusion

On July 9, 1990, several Laboratory scientists who had investigated phenomena purportedly demonstrating cold fusion were invited to discuss their observations. In light of some of the unusual events surrounding research on cold fusion, the *E&TR* asked these scientists to comment on a broad range of topics related to the idea of the scientific method as a working mechanism. Their answers reveal a pattern of developments over the past year in which the scientific method itself emerges as the ultimate arbiter.

Contact: Brian K. Balke (415) 423-5711, G. Bryant Hudson (415) 423-2947, Keith I. Thomassen (415) 422-9815, or Richard Van Konynenburg (415) 422-0456.

Using MeV Ions To Characterize and Modify Materials

The Laboratory has enhanced its capabilities for characterizing and modifying materials using MeV-ion-beam techniques with the addition of a 4-MV ion accelerator system. MeV ions can be used to perform quantitative, nondestructive analysis of materials by four ion-beam techniques: ion backscattering, particle-induced x-ray emission, ion-induced nuclear-reaction analysis, and forward-recoil spectroscopy. Simultaneous use of more than one of these techniques maximizes the information developed for various types of materials. In addition, we can modify materials using high-energy ion implantation and irradiation. In this article, we present the basic concepts involved in the interactions of ions with materials and in the four techniques, describe ion-beam modification procedures for implantation and irradiation, and give some examples of MeV-ion-beam characterization of materials at LLNL.

Contact: Ronald G. Musket (415) 422-0483.

END

DATE FILMED

12/28/90