Introduction to Materials and Bioscience Neutron-Scattering Research

Alan J. Hurd and Dale W. Schaefer

So confident were Manhattan Project scientists about their understanding of the nucleus that the gun concept for detonating a nuclear explosion was never tested before "Little Boy" was deployed against Japan in World War II. The implosion detonation concept, however, required testing at the Trinity Site in New Mexico on July 16, 1945. In the implosion bomb, conventional explosives drive the nuclear explosion by rapidly assembling and then compressing a plutonium "pit." Implosion was used in "Fat Man," the second and last nuclear device exploded in war. The issues leading up to Trinity involved not only the nuclear properties of plutonium but also its chemistry and metallurgy. Plutonium had to be manufactured, isolated, and purified; its phase behavior (Figure 1) had to be determined; and the desirable phases had to be stabilized (Hammel 1995 and 2000). Equally challenging problems regarding the behavior of conventional high explosives had to be solved. These issues fall in the realm of materials science, the study of the properties of condensed matter, for which neutron scattering is a unique and precise probe even though those properties are largely independent of the nucleus.

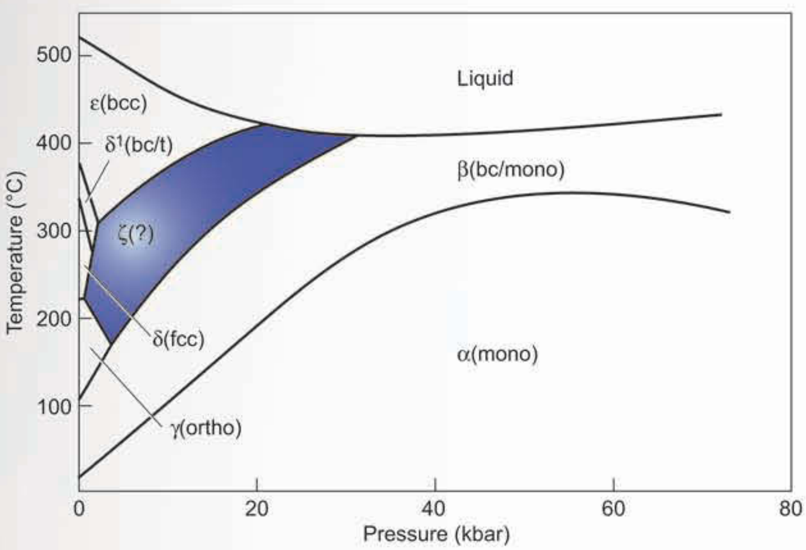

Figure 1. Plutonium at High Pressure and Temperature
The complicated phase diagram of plutonium was a major issue in working with the metal for the Trinity test. Just weeks before the test, the desirable δ-phase was stabilized by proper alloying. The ability to probe the properties of metals with high atomic weights in extreme environments is one of the important characteristics of neutron scattering. Indeed, much materials science remains to be done; even the structure of one high-pressure plutonium phase is still unknown.

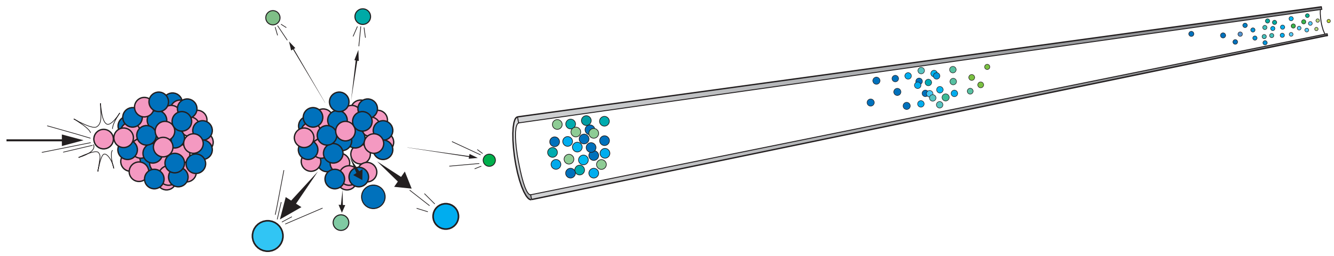

Figure 2. Spallation Neutrons
When a pulse of energetic protons (pink) hits a tungsten target, each proton that collides with a tungsten nucleus as in (a) causes the nucleus to release tens of neutrons with different energies, represented by different colors in (b). Neutrons traveling in a particular direction move along a beam line (c). The resulting pulse of neutrons is very short, but as the pulse travels down the beam line, the higher-energy neutrons travel faster, the pulse stretches out in space, and the arrival times of the neutrons serve to identify their energies and wavelengths.

The Manhattan Project illustrates that critical technologies are often limited by the availability of appropriate materials. This situation is unlikely to change. According to national studies of the last 30 years, the properties of 21st century materials must exceed those of today's materials by large margins. The projected mid-century "end-of-oil" challenge, for example, will elicit new hydrogen-storage materials, more-efficient solar cells, better nuclear fuels, and improved nuclear-reactor materials. In addition, the nuclear-weapon stockpile requires reliable prediction of materials aging to maintain deterrence without testing. Finally, the world faces emerging threats fueled by terrorism that demand significant materials advances. If history is a reliable guide, advances in materials research will be needed to meet these challenges.

Given the central role of materials science "in the beginning" and the importance of materials in emerging technologies, it is not surprising that materials science enjoyed an important position in the research portfolio of Los Alamos National Laboratory. Less obvious is the evolving role that subatomic particles, such as the neutron and proton, play in Los Alamos materials research.

Role Reversal

Conceived by Louis Rosen as a facility to explore nuclear structure, the Los Alamos proton accelerator was originally dubbed the Los Alamos Meson Physics Facility (LAMPF). LAMPF provided the proton beam for many discoveries in the realm of medium-energy nuclear physics, but eventually evolved into a source of protons and neutrons serving the needs of the materials science community at Los Alamos, at other Department of Energy laboratories, and now across the community of external users (see Figure 17 on page 16 of this volume). LAMPF was renamed the Los Alamos Neutron Science Center (LANSCE) in 1995.

As part of this evolution, the Lujan Neutron Scattering Center (Lujan Center) at LANSCE was built to study the properties of materials using the neutron as a probe. At the Lujan Center, protons are slammed into a tungsten target (Figure 2), and the resulting nuclear reactions produce copious quantities of neutrons that are then directed to 16 beam lines instrumented to explore the structure and dynamics of materials. At LANSCE, neutrons serve materials science as well as nuclear physics research.

Why Neutron Scattering?

The properties of materials are controlled by the positions and motions of atoms. The body of information regarding the position of atoms is called structure, and the information regarding motion is called dynamics. Although other techniques measure structure and dynamics, the neutron has a number of properties that make neutron scattering a unique probe for materials research.

First of all, neutrons are "poor, uneducated, and easy to command." Using moderators, devices that change the neutron energy spectrum, spallation neutrons can be cooled to match the structural length scales and the excitation energies of dynamics in materials. Although some techniques require thermal neutrons, the current emphasis throughout the world is on cold neutrons, which are useful for

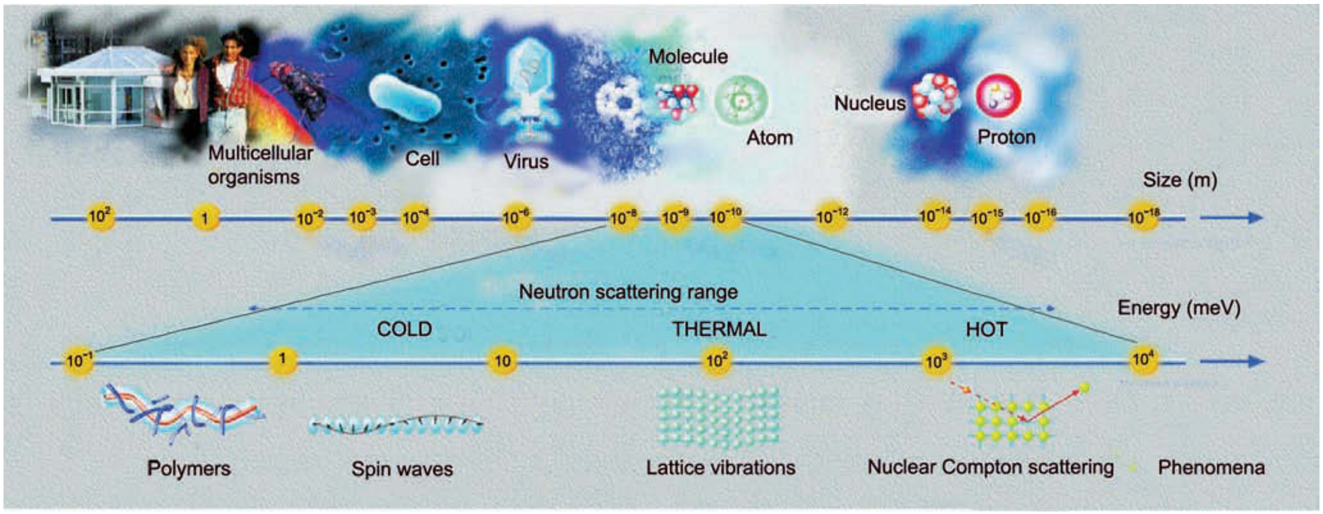

Figure 3. Neutron Energy Spectrum
A cold-neutron energy spectrum (the range is depicted on the energy axis) is best for scattering from polymers, large molecules, biological systems, and other soft materials. LANSCE plays a leading role in cold-neutron peak flux and instrumentation. Long-pulse technology will substantially enhance Lujan Center capabilities in soft matter and bio materials. **(This figure was adapted from "The European Spallation Source Project Report" 2002.)**

probing large-length-scale structures and low-energy excitations characteristic of soft matter, such as polymers, liquid crystals, colloids, and biological materials (Figure 3). In fact, the interest in cold neutrons and in long-pulse sources, which use cold neutrons efficiently, matches the rapidly growing interest in soft-matter research.

Many experiments can be accomplished only with neutrons. Because neutrons are more penetrating than x-rays, one can probe samples inside high-pressure vessels, refrigerators, and furnaces, or measure deeply buried structures in a bulk material (see the article "Plutonium under Pressure" on page 84 and Figure 1). Neutrons scatter more strongly from some isotopes than others of the same element, which makes isotopic labeling a major advantage as described below. Neutrons are also magnetic, and they are therefore particularly suited to the study of magnetism (see the article "Origins of Spin Coupling across Interfaces" on page 178). The predicted existence of magnetism in plutonium, for example, is important to the electronic structure of this metal and therefore to its equation of state, a very important factor in weapons physics. Low-energy neutrons match well the spectrum of excitations that underlie the physics of plutonium's electronic structure.

In 1986, the world learned about an entirely new phase of matter signaled by high-temperature superconductivity. Neutron scattering played a central role in understanding high-temperature superconductivity because, unlike x-rays, neutrons are adept at probing magnetic structure and crystal excitations and excel at probing compounds containing high atomic numbers. Nevertheless, the elucidation of high-temperature superconductivity is not yet completed after 20 years (see the article "Unraveling the True Atomic Structures of Exotic Oxides" on page 152).

Because the neutron scattering power of atoms is isotope dependent, labeling techniques are widely employed to sort out complex structures and dynamics (see the article "The Hydrophobic Effect—Why Do Raindrops Slide off Leaves?" on page 164). Hydrogen and deuterium, which are nearly invisible to x-rays, are easily located with neutrons.

Emerging Opportunities: Soft Matter and Bioscience

Starting with the 1975 National Academy of Sciences report "Materials and Man's Needs," the emergence of soft matter as a discipline was anticipated by numerous studies exploring the future direction of materials science. Encouraged by the National Nanotechnology Initiative and the remarkable progress in molecular biology over the last 5 years, soft matter currently dominates hiring in physical sciences at research universities across the world (National Science Board 2004).

The evolution of national security priorities at the Laboratory tracks the emergence of soft matter as a discipline. The traditional focus on metallurgy and condensed matter physics has shifted toward the chem-bio-radiological threats characterizing the period after the 9/11 terrorist attacks

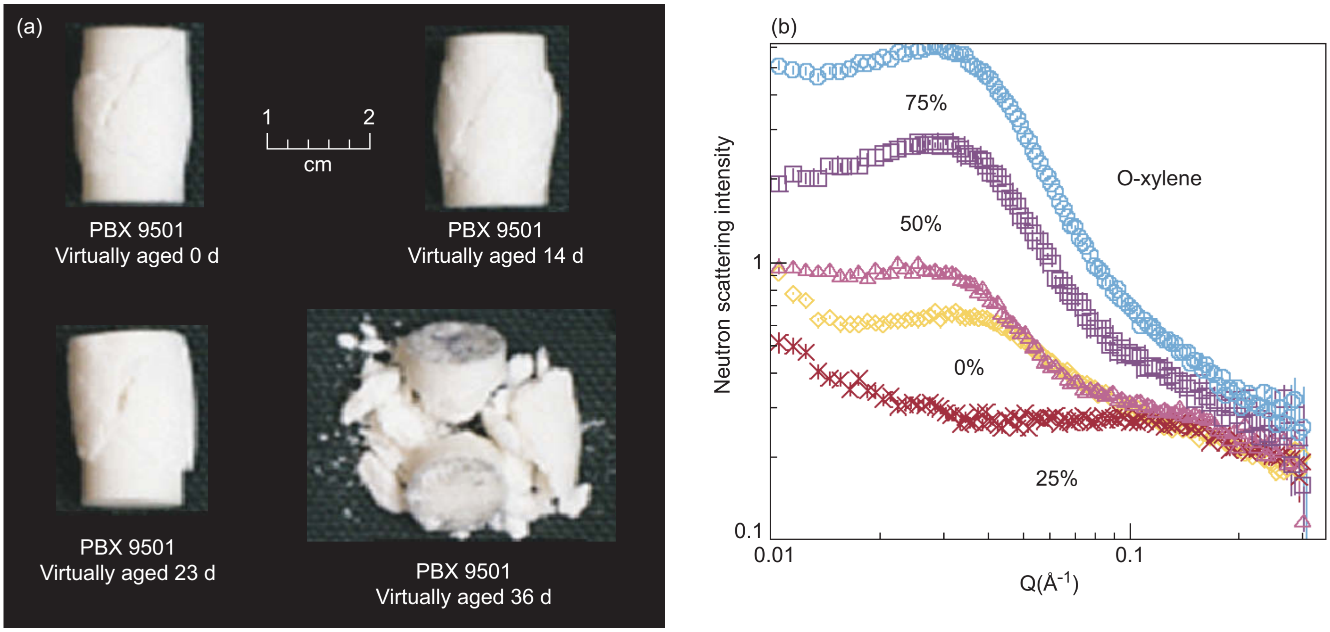

Figure 4. High Explosives Age

(a) The insensitive high explosive PBX 9501 exhibits degradation upon artificial, or virtual, aging. That is why PBX 9501 is being replaced by PBX 9502 and other formulations in the stockpile. Neutron scattering studies are being used to identify the keys to aging. (b) Estane™, a polymer binder, is the subject of intense research on aging. Ortho-xylene dissolves part of the Estane™ binder. By using a series (0% to 100%) of deuterated o-xylene, one changes the scattering contrast between the hard and soft domains without changing the chemistry or structure. At 25% deuterated o-xylene, the hard domains of Estane™ are seen to match. Future studies of aged Estane™ will be based on the power of contrast variation to look at hard and soft domains independently.

on the United States. The emphasis on maintaining the current nuclear stockpile and developing robust materials for refurbishment also evokes a host of new materials issues. The aging of high explosives in weapon systems, for example, is a topic of intense study, and neutron scattering allows researchers to distinguish physically different parts within these complex polymer molecules (Figures 4a and 4b). These new priorities underlie the growth of soft-matter research, including biosciences, at Los Alamos.

Nanotechnology is another distinguishable area of materials research enjoying worldwide expansion. The corresponding field of nanomaterials refers to matter organized on nanometer– (10^{-9} meter) length scales. Large-scale organization distinguishes nanomaterials from molecular materials, whose properties are determined by short-range molecular bonding.

The preferred synthesis path for soft nanomaterials is bottom-up self-assembly, whereby specific short-range interactions are engineered into complex precursor macromolecules. These short-range forces induce long-range order by cooperative physical interactions. Nanomaterials share many characteristics with soft materials in that the essential structural features occur on supramolecular scales and the corresponding dynamics are found at low energy.

Even weapons materials have nanoscale features important to their health and function. Inside neutron tubes, erbium tritide targets, when bombarded with accelerated deuterons, produce copious neutrons via a nuclear reaction. Because the tritium decays in timeto helium, which ruins the tube's vacuum, the target films are designed to retain helium. When retention is low, tube lifetime is low. Owing to its high acuity for light elements, neutron scattering has proved to be effective in determining the mechanisms of helium retention and release. Figure 5 shows small-angle, neutron scattering results that indicate helium buildup along preferred lattice directions. Strategies to adjust the microstructure (grain boundaries) can then be proposed to mitigate the aggregation of helium.

The recurring theme in this discussion of soft matter and biomaterials is large-scale structure. This theme elicits a new perspective on neutron-scattering instrumentation. The long-pulse spallation source, for example, is a new concept recently demonstrated at LANSCE that meets the need to measure structure and dynamics of soft matter and to do bioscience research.

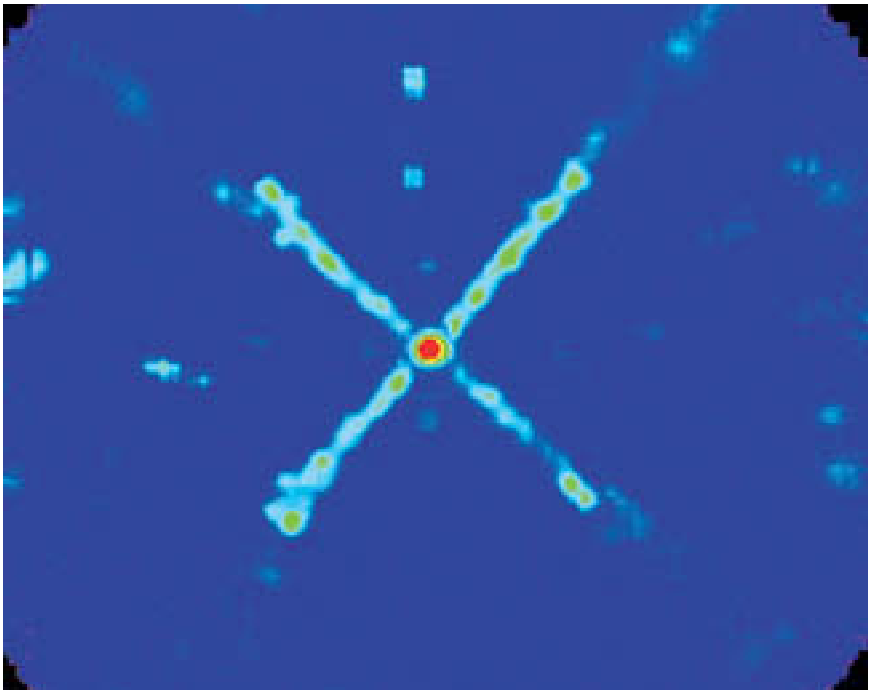

Figure 5. Scattering in Erbium Tritide
This remarkable pattern of small-angle neutron scattering demonstrates that helium released by the radioactive decay of tritium collects preferentially along planes in the lattice. This mechanism limits helium retention by films, thereby shortening neutron tube lifetime. **(This image is courtesy of James Browning of Sandia National Laboratories, Albuquerque, NM.)**

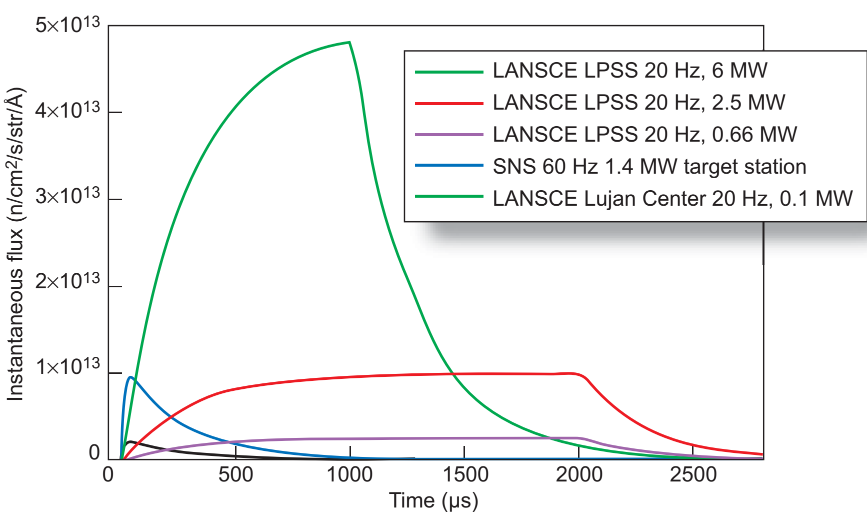

Figure 6. Cold-Neutron Line Shapes for Various Pulsed Sources
In long-pulse neutron sources, proton energy is deposited in the spallation target over a much longer period, which mitigates target degradation. The integrated neutron flux of the LPSS pulses is fully used in applications requiring moderate wavelength resolution, such as small-angle scattering. In applications requiring short-wavelength resolution (for example, medium- and high-resolution diffraction), pulse-shaping choppers will be used to produce pulses with variable lengths, including shorter ones than those available at the SPSS. The 6-MW calculation (green curve) represents the ultimate LPSS potential at LANSCE. **(Ferenc Mezei, Los Alamos National Laboratory, personal communication, June 2005.)**

Long-Pulse Spallation Technology

The demonstration of neutron production from proton beams by spallation in the 1970s offered new hope for a source more intense than fission reactors. Using time-of-flight techniques, pulsed neutrons can be sorted by energy and can be used one by one. In contrast, energy discrimination at steady-state sources requires filtering out an overwhelming fraction of the neutrons.

The challenge for the future is to build neutron sources that combine the efficiency of pulsing with enhanced time-average flux (see Figure 16 on page 15 of this volume). This goal can be achieved using long-pulse spallation source (LPSS) technology. The long-pulse technique circumvents the limitations of the short-pulse spallation source (SPSS) approach, which will be pushed close to its technical limit at the Spallation Neutron Source (SNS) currently under construction at Oak Ridge National Laboratory. The beauty of the LPSS is that the instruments most useful for soft matter and bioscience research can achieve much higher data rates while the instruments optimized for hard materials are only slightly compromised.

Short-pulse spallation sources are limited by the proton beam energy that can be delivered in a single proton pulse. The limit is imposed by both accelerator technology (space charge limits in ring accelerators) and target degradation. In linear accelerators, long, energetic proton pulses can be produced that are far from the space charge limit. Moreover, by comparison with the SPSS, long pulses avoid the target material problems because energy deposition is spread over a much longer time (see Figure 6). Such pulses still afford reasonable velocity definition for long-wavelength neutrons.

The above considerations imply that LPSS technology can provide neutrons with more than an order of magnitude higher brilliance than the neutrons produced by the SPSS. LPSS can also surpass continuous reactor sources in terms of time average flux and provide superior performance in most applications of neutron beam research.

Conclusions

There is little doubt that basic and applied research in materials and bioscience will define the Los Alamos science portfolio for the foreseeable future regardless of whether the Laboratory's mission emphasizes nuclear weapons, threat reduction, or energy. Solving the structure property puzzle for 21st century materials will require characterization tools that keep pace with advances in synthesis. For 60 years, neutron scattering has been an indispensable technique for materials characterization. In the budding world of nanomaterials, biomaterials, and soft materials, neutron sources optimized for the study of supramolecular structures and collective motions are required. LPSS technology matches this requirement by exploiting long-wavelength low-energy neutrons. Compared with short-pulse instruments, LPSS instruments will yield large performance gains in the study of soft matter and biomaterials.

Further Reading

Commission on Engineering and Technical Systems, National Research Council. 2000. *Summary Record of the Workshop on Polymer Materials Research*: *August 30–31*, *1999*, *Woods Hole*, *Massachusetts*. Washington, DC: The National Academies Press.

Committee on Advanced Energetic Materials and Manufacturing Technologies, National Research Council. 2004. *Advanced Energetic Materials*. Washington, DC: The National Academies Press.

Committee on Alternatives and Strategies for Future Hydrogen Production and Use, National Research Council, National Academy of Engineering. 2004. *The Hydrogen Economy*: *Opportunities*, *Costs*, *Barriers*, *and R&D Needs*. Washington, DC: The National Academies Press.

Committee on Condensed-Matter and Materials Physics, National Research Council. 1999. *Condensed-Matter and Materials Physics*: *Basic Research for Tomorrow's Technology*. Washington, DC: The National Academies Press.

Committee on Materials Research for Defense After Next, National Research Council. 2003. *Materials Research to Meet 21st Century Defense Needs*. Washington, DC: The National Academies Press.

Committee on National Laboratories and Universities, National Research Council. 2005. *National Laboratories and Universities*: *Building New Ways to Work Together—Report of a Workshop*. Washington, DC: The National Academies Press.

Committee on Science, Engineering, and Public Policy. 1975. *Materials and Man*'*s Needs*: *Materials Science and Engineering—Volume II*, *The Needs*, *Priorities*, *and Opportunities for Materials Research*. Washington, DC: The National Academies Press.

Committee of Soldier Power/Energy Systems, National Research Council. 2004. *Meeting the Energy Needs of Future Warriors*. Washington, DC: The National Academies Press.

ESS Council. 2002. *The European Spallation Source Project*. Complete Edition (Volumes I–IV), ISBN 3-89336-299-1. Germany: Druckerei Plump OHG.

ESFRI Working Group on Neutron Facilities. 2003. *Medium to Long-Term Future Scenarios for Neutron-Based Science in Europe* [online]: http://neutron.neutron-eu.net/n_documentation/n_reports

Hammel, E. 1995. Plutonium Metal: The First Gram. *Los Alamos Science* **23**: 162.

———. 2000. The Taming of "49"—Big Science in Little Time. *Los Alamos Science* **26**: 48.

National Science Board. 2004. *Science and Engineering Indicators 2004*. Two volumes. Arlington, VA: National Science Foundation (Volume 1, NSB 04-1; Volume 2, NSB 04-1A).

Organizing Committee for the Workshop on Energy and Transportation, Committee on Challenges for the Chemical Sciences in the 21st Century, National Research Council. 2003. *Energy and Transportation*: *Challenges for the Chemical Sciences in the 21st Century*. Washington, DC: The National Academies Press.

Proceedings of the 2000 National Materials Advisory Board Forum, National Research Council. 2001. *Materials in the New Millennium*: *Responding to Society*'*s Needs*. Washington, DC: The National Academies Press.

Solid State Sciences Committee, National Research Council. 1987. *Advancing Materials Research*. Edited by P. A. Psaras and H. D. Langford. Washington, DC: The National Academies Press.

U.S. Department of Energy's Environmental Management Science Program, National Research Council. 2000. *Research Needs in Subsurface Science*. Washington, DC: The National Academies Press.