

# RTNS-II 1984 Annual Report

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## About the Cover

We are analyzing miniature tensile specimens irradiated at the RTNS-II to study the mechanical property changes that are induced by 14-MeV neutrons. The dimensions of the specimen shown are  $12.7 \times 2.54 \times 0.25$  mm. The two graphs appearing beneath the specimen plot tensile data for annealed Marz-grade copper irradiated at 90°C. The graph to the left traces stress-strain curves for unirradiated copper (lower curve) and for copper irradiated to a fluence of  $1.4 \times 10^{18}$  n/cm<sup>2</sup>. The bottom right graph shows how copper's 0.2%-offset yield stress changes as a function of 14-MeV-neutron fluence. To study the effects of the difference in neutron energies, we are now comparing the tensile property changes of materials irradiated here at elevated temperatures with those irradiated under the same conditions at the Omega West Reactor at Los Alamos National Laboratory.

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## Foreword\*

This is the third annual report summarizing irradiation experiments at RTNS-II. It covers calendar year 1983, and includes reports of all irradiation results, both fusion and non-fusion related. These comprise both secondary (or "add-on") and primary irradiations.

Each summary article has been submitted by the investigator and has been altered only to meet the style and format requirements of this report. Previous information concerning irradiations and the facility are contained in the RTNS-II 1982 Annual Report (UCID-19837-82), RTNS-II 1983 Annual Report (UCID-19837-83), and the Guide for Experimenters (LLNL-M-094 Rev. 1, March, 1982).

Dale W. Heikkinen  
Deputy Facility Manager, RTNS-II

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\*Work performed under the auspices of the U.S. Department of Energy and the Japan Ministry of Education, Science and Culture (Monbusho) by the Lawrence Livermore National Laboratory under contract number W-7405-ENG-48.

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## Overview—The RTNS-II Facility

Early in 1982 a unique collaboration began between the governments of Japan and the United States. At that time the U.S. Department of Energy (DOE) and the Japan Ministry of Education, Science, and Culture (Monbusho) agreed jointly to support the operation and development of RTNS-II, and to share in its utilization.

RTNS-II was built to provide a deuterium-tritium neutron source for the study of fusion neutron effects. In the quest to apply fusion to commercial power production, the specific mission of RTNS-II is threefold:

- To acquire direct engineering data for near-term confinement experiments and for materials that will see moderate neutron dose in future reactor systems.
- To measure production rates of transmutants and to develop appropriate radiation-resistant instrumentation for fusion systems.
- To study the radiation-induced property changes caused by fusion neutrons.

RTNS-II comprises two independent sources of 14-MeV neutrons. Deuterium ions are extracted from the ion source at 30 keV.  $D^+$  ions are selected by a 90-deg bending magnet. The air-insulated terminal is held at roughly 340 keV by a Cockroft-Walton power supply. We now deliver 130 mA of roughly 370-keV deuterons to the target in a beam spot size about 1 cm in diameter. This provides a neutron source strength approaching  $4 \times 10^{15}$  n/s. The nominal operating schedule is 24 hours a day, 5 days a week.

The rotating target has a titanium-tritide coating on a copper alloy substrate. This material would thermally decompose at an unacceptable rate if its temperature were allowed to rise above about 300°C. Thus, to limit the peak temperature produced by beam heating, the target rotates at 5000 rpm and is intensively cooled by chilled water flowing through internal channels. An air-levitated, differentially pumped vacuum seal permits target-assembly rotation with negligible leakage of air into the vacuum system.

Meeting the materials challenges of fusion systems requires that we use a broad array of research tools. In this context, RTNS-II has a unique role. It is the world's only 14-MeV-neutron source dedicated exclusively to materials research.

## Neutron Monitor Calibration

D. W. Heikkinen

Lawrence Livermore National Laboratory, Livermore, CA

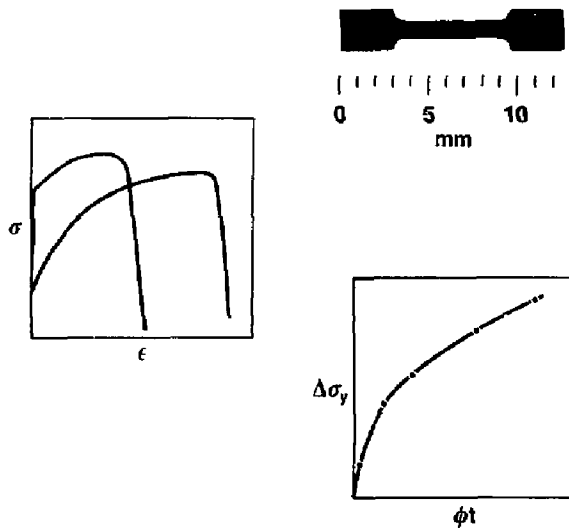
To provide instantaneous and cumulative data on neutron production for the RTNS-II neutron sources, neutrons are monitored using proton-recoil counters and ionization chambers. These data also provide the time history of an irradiation which is useful for obtaining neutron fluences from dosimetry.

The neutron production monitors are periodically calibrated by placing niobium dosimetry foils on the front surface of the individual monitors. Repeated calibrations have shown no large changes in the calibration constants.

All neutron monitor calibration records are maintained at the RTNS-II facility.

# RTNS-II

## Japan-Initiated Fusion Experiments



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# 14-MeV Neutron Irradiation Hardening in Metals, Alloys, and Other Materials

Katsunori Abe

The Research Institute for Iron, Steel, and Other Metals  
Tohoku University, Sendai 980, Japan

A total of 2300 specimens, including tensile specimens and transmission electron microscopy (TEM) disks, were irradiated at room temperature by the RTNS-II. These specimens, which have been supplied from sixteen material research groups in Japan, include: (a) pure metals and their alloys (Al, Al-Si, Ti, V, V-C, VTi, V-Fe, V-Co, V-B, Fe, Ni, Ni-Si, Cu, Zr, Nb, Mo, Ag, Au); (b) fusion-oriented practical alloys (Al-Mg-Li, SUS 304, SUS 316, JPCA, Ferritics, V-20Ti, TZM); (c) amorphous metals ( $Pd_{80}Si_{20}$ ,  $Fe_{80}B_{20}$ ,  $Fe_{78}Si_{10}B_{12}$ ,  $Zr_{70}M_{30}$ ,  $Zr_xCu_{100-x}$ ); and (d) polyimide film (KAPTON). Three irradiations over a period of 11 weeks produced maximum fluences of  $6 \times 10^{18}$  n/cm<sup>2</sup> in TEM disks and  $5 \times 10^{18}$  n/cm<sup>2</sup> in tensile specimens. Duplicate sets of specimens were irradiated for fusion-fission correlation study at the Kyoto University Reactor (KUR).

Neutron-irradiation research at LLNL can be divided into three main areas:

- (i) Irradiation and Testing Apparatus.
- (ii) Irradiation Hardening in Metals and Alloys.
- (iii) Irradiation Damage in KAPTON polyimide.

A summary of our progress in these three areas follows.

## Miniature Specimen Material Testing

A universal testing machine with temperature controller, a data analyser, and miniature specimen grips for tensile-, 3-point bend-and bulge-test were installed at LLNL. We used these facilities to correlate bend properties and microhardness with tensile properties for two metals, Mo and TZM, each one having been exposed to its own level of heat treatment and neutron fluence. This correlation enabled us to produce a wide range of stress levels.

## Irradiation Hardening in Metals and Alloys

Hardening by 14-MeV neutrons depends strongly on materials and their treatment. These may be divided into three groups:

- (i) Negligible hardening: Al, Al-Mg-Li alloy, and amorphous alloys.
- (ii) Small hardening (less than 50% hardening at  $3 \times 10^{18}$  n/cm<sup>2</sup>): V, Mo, TZM, Ferritics, Fe-9Cr and Cu-Prop.
- (iii) Large hardening (greater than 50% hardening at  $3 \times 10^{18}$  n/cm<sup>2</sup>): Fe, Ni, Au, Cu, SUS 316 (JPCA).

## Irradiation Damage of Kapton Polyimide Film

Because of its relatively high resistance to radiation damage, polyimide film may be used as an insulator for the fusion project's superconducting magnet. We measured the mechanical property changes that were included in KAPTON polyimide film by 14-MeV neutrons and  $\gamma$ -rays using the miniature tensile technique [1], and found that elongation decreases severely with fluences above  $10^{17}$  n/cm<sup>2</sup>. Changes in optical absorption correspond well with elongation changes. Fusion neutrons cause 70 $\times$  more damage than the equivalent  $\gamma$ -ray dose.

## Reference

- [1] K. Abe, K. Saneyoshi, C. M. Logan, and F. W. Clinard, Jr., "14-MeV-Neutron Irradiation Damage of KAPTON Polyimide Film," ICFRM-1 (1984, Tokyo), pp. 4-43.

## High Fluence Cryotransfer Experiment

H. Matsui and S. Iwasaki  
Tohoku University, Sendai 980, Japan

M. W. Guinan and J. S. Huang  
Lawrence Livermore National Laboratory, Livermore, CA

We irradiated a capsule containing 30 electrical-resistivity samples, 40 positron-annihilation samples, 1200 transmission electron microscopy (TEM) foils, 1500 bend-test samples, and 140 tensile samples at 20 K from October 18 to November 8, 1984. The neutron fluence in the capsule varied from  $3.5 \times 10^{16}$  to  $1.4 \times 10^{18}$  n/cm<sup>2</sup>. Following the irradiation, we transferred the sample capsule to a liquid nitrogen storage bath where, without warming above 77 K, the capsule was disassembled and all dosimetry foils were removed. The specimens were then transferred to a long-term LN storage dewar.

The experiment examined specimens supplied by 12 additional groups in Japan, representing Hiroshima, Hokkaido, Kyushu, Nagoya and Tohoku Universities, and the University of Tokyo. Included were: (a) pure metals and alloys of Fe, Ni, Mo, V, Cu, Al, Ti, Ag, Nb, Mg, Zr, and Au; (b) austenitic and ferritic steels; (c) silicon and germanium semiconductors; and (d) amorphous metals.

After a short cooling period, samples for both bend and tensile tests were transferred without warming to a measurement cryostat. Mechanical-properties tests were carried out at temperatures ranging from 4.2 K to room temperature.

Only a few preliminary tests were completed in 1984 to validate the transfer operations, and the results of these tests will appear in journals in late 1985. For a more detailed discussion of this work, see Hideki Matsui, "Mechanical Properties of Metals and Alloys Irradiated with 14-MeV Neutrons at Low Temperatures," in the 1984 *Annual Research Report of Japanese Contributions for Japan-US Collaboration on RTNS-II Utilization*, (Monbuscho, Japan Fusion Cooperation Program, March, 1985), pp. 55-75.

## Low Fluence Cryotransfer Experiment

Y. Shimomura

Faculty of Engineering, Hiroshima University, Shitami, Saijō-chō, Higashihiroshima City 724, Japan

M. W. Guinan

Nuclear Chemistry Division, Lawrence Livermore National Laboratory, Livermore, CA

A capsule containing 28 electrical resistivity samples, 46 positron-annihilation samples, and 835 transmission electron microscopy (TEM) foils was irradiated at 20 K from March 12 to March 17, 1984. The neutron fluence in the capsule varied from 0.6 to  $12.8 \times 10^{20}$  n/m<sup>2</sup>. Following the irradiation, the sample capsule was transferred to a liquid nitrogen storage bath. Without warming above 77 K, the capsule was disassembled, its dosimetry foils were removed, and the specimens were then transferred to a long-term LN storage dewar.

The experiment examined specimens supplied by 20 additional investigators in Japan, representing Hokkaido, Kyoto, Hiroshima and Kyushu Universities, and the Universities of Tokyo and Osaka. Included were: (a) pure metals and alloys of Fe, Mo, V, Ni, Au, Ta, Ti, Al, Cu, Zr, Ag, Pt, and Nb; (b) ferritic and austenitic steels; (c) germanium, silicon, and gallium arsenide semiconductors; (d) Cu<sub>3</sub>Au, Nb<sub>3</sub>Sn, Nb<sub>3</sub>Ce, V<sub>3</sub>Ge ordered alloys; and (e) silicon carbides and nitrides.

Specimens were successfully shipped to Japan at LN temperatures and subsequently transferred without warming to the positron-annihilation and TEM stages. TEM transfers were also made at LLNL.

At this time only some preliminary TEM observations have been presented at the *First International Meeting on Fusion Reactor Materials* in Tokyo, December 3-6, 1984. This paper has been submitted to *J. Nucl. Mat.* by Y. Shimomura, M. W. Guinan, and M. Kiritani, under the title "Low Temperature D-T Neutron Irradiation and Cryotransfer Observation of Cascade Defects in Metals."



# Secondary Defect Formation in Copper by 14-MeV Neutron Irradiation and Its Effect on Microstructure Evolution

N. Yoshida, Y. Akashi, and K. Kitajima

Research Institute for Applied Mechanics, Kyushu University, Kasuga 816, Japan

M. Kiritani

Faculty of Engineering, Hokkaido University, Sapporo 060, Japan

One of the typical features of 14-MeV-neutron irradiation is that the average primary knock-on energy reaches a few hundred keV in most materials, so that the high transfer energy of a single collision creates a cascade of several hundreds of point defects in a small region. The defects are successively rearranged and some of them form clusters. Though it has long been recognized that the rearrangement of a cascade is essential for damage-structure evolution under energetic particle irradiation (such as irradiation by 14-MeV neutrons), up to now its fundamental aspects have not been investigated well enough to understand the atomic processes of damage-structure evolution.

The purpose of the present work, then, is to reveal the rearrangement processes of lattice defects in a high-energy (14-MeV-neutron) cascade, and to understand the influence of such a cascade on microstructure evolution.

As a way of accomplishing this objective, we used the RTNS-II to irradiate pure Copper specimens at 25, 200, and 400°C at doses of  $3.6 \times 10^{18}$  n/cm<sup>2</sup>. We then examined the resulting damage structure by means of transmission electron microscopy.

We found that, at 25 and 200°C, stacking fault tetrahedra (SFT), partially dissociated Frank loops, vacancy aggregates, and interstitial loops were nucleated by cascade collapse. All of these defects have a tendency to group together, with as many as 10 defects forming a single cluster. Because SFT are very stable under irradiation, excess interstitials (corresponding to the vacancies retained in the SFT) are accumulated in the matrix and form their own clusters. Interstitial loops that nucleate near a dislocation grow preferentially by absorbing the interstitials as they migrate toward the dislocation.

It is interesting to note that voids were created by dose irradiations as low as  $1.0 \times 10^{18}$  n/cm<sup>2</sup>. The large number of vacancies retained in each void suggests that the voids have grown under irradiation by absorbing free vacancies. This process will play a very important role in void swelling at high doses.

## Defect-Structure Evolution after Radiation Damage from DT Fusion Neutrons

M. Kiritani

Faculty of Engineering, Hokkaido University, Sapporo 060, Japan

We have irradiated various metals at the RTNS-II and observed the resulting defect structures. Our purpose was to obtain a unified understanding of the defect-structure evolution associated with those irradiations which generate large cascades. We measured the maximum separation of interstitial atoms from the vacancy-rich zone, and found that (1) vacancy-type defect clusters create groups that reflect the damage caused by sub-cascades, and (2) high resolution stereo-electron microscopy discloses the three-dimensional configuration of these sub-cascades. The effective collision cross-section needed to produce defect clusters was estimated, and the damage efficiency was obtained. It is possible to understand the roles of free interstitials released from cascade zones, the elimination of vacancy-clustered defects, and the formation of their own clusters, by comparing the defect structures of bulk specimens with those of thin foils that were removed from the bulk after irradiation.

This work was done in collaboration with Y. Shimomura and N. Yoshida. The low-temperature irradiation was performed in collaboration with M. W. Guinan. The author has given a more detailed description of this work at the *First International Conference on Fusion Reactor Materials* held in Tokyo (December, 1984). That report will be published in *J. Nucl. Mat.*

## 14.8-MeV Neutron-Induced Activation in Some Metals and Practical Alloys

Keiji Saneyoshi

Department of Energy Sciences, Tokyo Institute of Technology, Nagatsuta, Yokohama 227, Japan

Katsunori Abe

The Research Institute for Iron, Steel, and Other Metals, Tohoku University, Sendai 980, Japan

We have measured the DT neutron cross sections for 41 reactions involving 15 elements that are of interest for fusion reactors. The measurement was performed using the activation method, with an error of less than 10%. An intense neutron source and simultaneous irradiation reduced the statistical and systematic errors and lowered the irradiation time as well. We developed a computer code that uses these cross-section data to calculate dose rates from some practical irradiated alloys. The calculation agrees very well with experimental results.

## Effects of 14-MeV Neutron Irradiation on SiC Fibers

K. Okamura, T. Matsuzawa, and M. Sato

The Oarai Branch, RIISOM, Tohoku University, Oarai, Ibaraki-ken 311-13, Japan

K. Sumita and T. Iida

Department of Nuclear Engineering, Osaka University, Yamada-oka, Suita-shi, Osaka 565, Japan

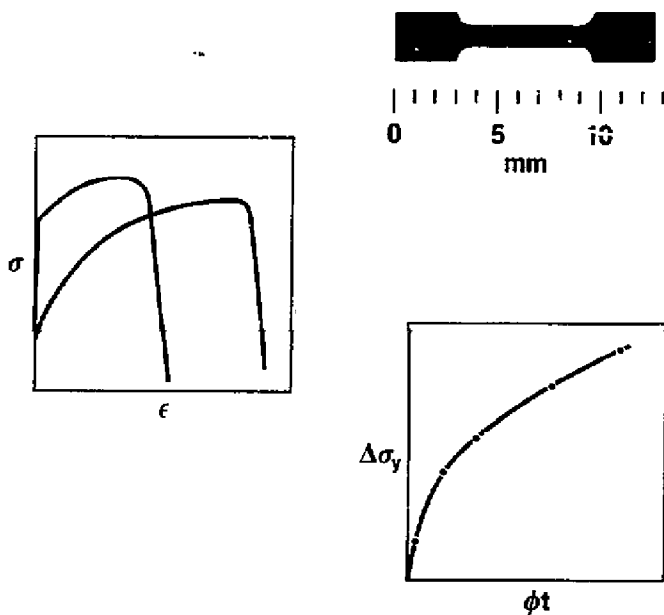
SiC is one of the candidate materials for the first-wall of fusion reactors [1]. To examine the feasibility of SiC fibers for this purpose, we irradiated them with 14-MeV neutrons. Two types of fibers were obtained, one by heating the polycarbosilane precursor fiber at 1000°C, the other at 1300°C, in an N<sub>2</sub> gas flow. The 1000°C fiber is in the amorphous state, and the 1300°C fiber is in the microcrystalline β-SiC state.

Both fibers were irradiated at RTNS-II (in the air at room temperatures) to fluences of about  $4 \times 10^{17}$  n/cm<sup>2</sup>. We made tensile-strength tests and x-ray diffraction analyses of the neutron-irradiated SiC fibers so that we could study the neutron-irradiation effects.

The results of these tests are given in the *1984 Annual Research Report of Japanese Contributions for Japan-US Collaboration on RTNS-II Utilization* (March, 1985), p. 106.

### Reference

- [1] L. H. Rovner and G. R. Hapkins, *Nucl. Technol.* **29** (1976), p. 274.



## Permeation of $^{13}\text{N}$ -Labeled Gas Molecules Through Semi-Permeable Membranes

R. A. Jalbert

Los Alamos National Laboratory, Los Alamos, NM

The Los Alamos National Laboratory is developing an instrument for the Princeton Plasma Physics Laboratory that will both measure and differentiate the concentration of tritium in elemental and oxide forms. It will also analyze the concentration of the activated air that will be produced by the D-T neutrons of the Tokamak Fusion Test Reactor. The instrument will use semipermeable membrane driers to separate HTO from the other radioactive gases and will incorporate a catalyst to oxidize the HT to HTO. Tests at RTNS-II have indicated that the permeability of the  $^{13}\text{N}$  that was produced by D-T neutrons (presumably in the form of  $^{13}\text{NO}_x$ ) passing through the several driers tested was less than 0.1% of the permeability of HTO, which included Perma Pure Driers using Dupont Nafion capillary tubing along with commercial kidney dialyzers. Final testing of the complete, assembled instrument at RTNS-II is planned for March, 1985, before we ship the instrument to Princeton.

A description of the instrument will be given at the *American Nuclear Society National Topical Meeting* at Dayton, Ohio (April-May, 1985).

## Spinel Swelling after Low-Dose Neutron Irradiation

W. A. Coghlan and F. W. Clinard, Jr.

Los Alamos National Laboratory, Los Alamos, NM

N. Itoh

Nagoya University

L. R. Greenwood

Argonne National Laboratory, Argonne, IL

As part of a fission- and fusion-neutron correlation study of ceramics, we irradiated single-crystal spinel ( $\text{MgAl}_2\text{O}_4$ ) with fast-fission neutrons up to doses of  $8 \times 10^{18}$  n/cm<sup>2</sup> ( $E > 0.1$  MeV). Irradiations were carried out in the Omega West Reactor at roughly 50°C. We found that swelling effectively saturated at about  $2 \times 10^{18}$  n/cm<sup>2</sup>, corresponding to a damage level of only about  $2 \times 10^{-3}$  dpa. These results imply that defects begin to interact by recombination, aggregation, or both at low damage levels in spinel. We are using rate equations to determine which of these kinetic processes dominates. Results to date show that the observed swelling is consistent with the number of surviving defects calculated from these limiting cases, if swelling per Frenkel pair is taken to be 1 at. vol. Measurements of swelling from 14-MeV-neutron irradiation will be made at RTNS-II for comparison with these findings.

This work has been submitted for publication in the *Seventh Annual Progress Report on Special Purpose Materials for Magnetically-Confined Fusion Reactors* (DOE/ER 0113/4).

## **Hardening of Instrument Channel Components for Fusion Radiation Environments**

**C. P. Cannon**

**Hanford Engineering Development Laboratory, Richland, WA**

Ceramic-to-metal seals are essential to many instrument channels. They are usually required to maintain hermeticity to  $10^{-9}$  atm-cc/He, and their electrical properties must likewise remain unimpaired. Experience has shown that this type of component is vulnerable in hostile radiation environments, such as will be encountered in fusion applications. This experiment is designed to answer several key questions regarding fabrication parameters of ceramic-to-metal seals. Our purpose is thus to extend the base technology from which to design seals for 14-MeV neutron environments.

A test package containing specially fabricated ceramic-to-metal seals has been prepared and is now being irradiated in RTNS-II. The seals were designed to evaluate the effects of the 14-MeV flux on the following design parameters:

- (i) ceramic purity, especially as influenced by trace impurities.
- (ii) metallization paint.
- (iii) selected geometrical factors.

Electrical properties affected by the irradiation are also being evaluated. The test objective—irradiation to  $3 \times 10^{16}$  n/cm<sup>2</sup>—is scheduled for completion by May, 1985.

## **Fusion-Neutron Irradiation of Nb<sub>3</sub>Sn-Ti Alloy Superconductors**

**C. L. Snead, Jr.**

**Applied Science Department, Brookhaven National Laboratory (BNL), Brookhaven, NY**

**M. W. Guinan**

**Nuclear Chemistry Division, Lawrence Livermore National Laboratory, Livermore, CA**

The purpose of this experiment is to make an initial assessment of the influence of titanium additions on the irradiation response of five Nb<sub>3</sub>Sn alloys. One set of alloy samples will be irradiated at RTNS-II, and an identical set will be irradiated to equivalent damage energies at the High Flux Beam Reactor (HFBR) at BNL.

The five alloy superconductors (which contained 0, 0.5, 1.0, 2.0, and 3.0 at. % titanium) were prepared by the "bronze process." We formed the Nb<sub>3</sub>Sn layers through heat treatment at 725 °C for 120 h in a vacuum that was voided to better than  $10^{-5}$  torr. Twelve samples of each composition were separated into six packets (two wires of each composition) and then placed in a single irradiation package so that, at the end of the room-temperature irradiation, we might achieve fluences of 3.2, 1.6, 0.8, 0.4, 0.2, and  $0.1 \times 10^{16}$  n/cm<sup>2</sup>.

The irradiation was carried out with that of C. Cannon and R. Flükiger (see p. of this annual report). At the end of 1984, about one-third of the target dose had been achieved. We expect to finish the irradiation by March or April of 1985.

Following irradiation, measurements of  $T_c$  will be carried out at LLNL on each of the sixty samples. After receiving the samples, Dr. Snead will measure their critical current density at 4.2 and 1.8 K at fields up to 24 T using the facilities of the National Magnet Laboratory at MIT.

## Measurement of 14-MeV Cross Sections

R. K. Smither and L. R. Greenwood  
Argonne National Laboratory, Argonne, IL

D. G. Doran and H. L. Heinisch  
Hanford Engineering Development Laboratories, Richland, WA

The object of this work is to measure the following reaction cross sections at 14.8-MeV-neutron energy:  $^{94}\text{Mo}(n,p)^{94}\text{Nb}$ ,  $^{94}\text{Mo}(n,2n)^{93}\text{Mo}$ ,  $^{63}\text{Cu}(n,p)^{63}\text{Ni}$ , the sum of  $^{54}\text{Fe}(n,2n)^{53}\text{Fe}(\beta^-) \rightarrow ^{53}\text{Mn}$  and  $^{54}\text{Fe}(n,d)^{53}\text{Mn}$ . All of these reactions produce stable isotopes that could worsen the waste disposal problems of a fusion reactor. Little or no information is presently available on these cross sections, and measurements are complicated by the long lifetimes and the lack of characteristic gamma rays in the decay chain.

In this work a relatively new method, accelerator mass spectrometry (AMS), will be used to measure the concentration of the newly formed isotope in the sample. AMS makes a direct count of the rare atoms in a particle beam and compares this rate with that of a current beam of the normal isotope. Such an approach circumvents the usual problems associated with long-lived isotopes and with a shortage of countable gamma rays. We used separated isotope material (enriched to 92%  $^{94}\text{Mo}$ ) for the molybdenum sample in order to reduce the interference from other isotope products. All the other samples consisted of natural abundance.

The irradiations at RTNS-II have been completed and the samples are now being prepared for shipment to Argonne National Laboratory where the mass spectroscopy will be completed.

## Low-Exposure Spectral-Effects Experiment

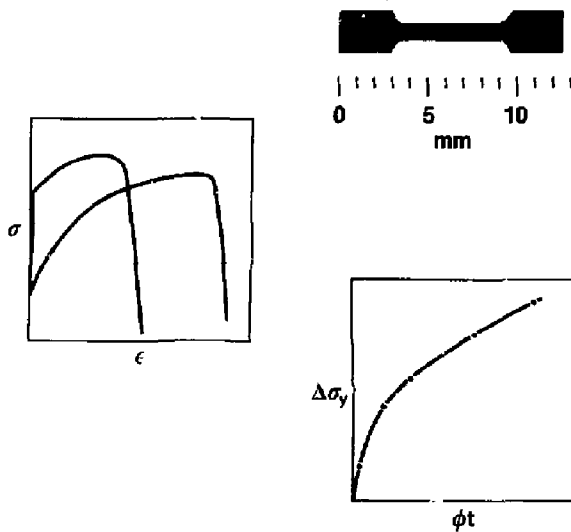
H. L. Heinisch  
Westinghouse Hanford Company, Richland, WA

We are planning to irradiate several miniature flat tensile specimens of pure metals and alloys at elevated temperatures in RTNS-II and the Omega West Reactor at Los Alamos National Laboratory. Our purpose is to study the effects of two very different neutron spectra on tensile properties and microstructures. The materials, Marz-grade copper, Marz-grade iron, AISI 316 stainless steel, and A302B pressure-vessel steel, constitute a representative sample of fusion-relevant face-centered cubic and body-centered cubic metals and alloys. They were chosen for their anticipated tensile sensitivity to very low exposures.

The first irradiation we performed was the joint US-Japan RTNS-II Experiment #9. Specimens were irradiated simultaneously at 90 and 290°C using the HEDL dual-temperature vacuum-insulated furnace. On completion of the irradiation (September 23, 1984) we achieved a peak fluence of  $2.4 \times 10^{18}$  n/cm<sup>2</sup>. The prime volume of the furnace (i.e., the first 6 mm, over which the flux drops by about 65%) was occupied by the tensile specimens for the HEDL Spectral-Effects Experiment, and the remaining volume was occupied by specimens of several other Japanese and US experimenters.

Tensile specimens for this experiment were also irradiated in the Japanese room-temperature irradiation. The miniature tensile specimens from both the Japanese and American irradiations are now being tested.

A more complete description of the Low-Exposure Spectral-Effects Experiment can be found in the *DAFS Quarterly Progress Report* (January-March, 1984), DOE/ER-0046/17, p. 76.



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## **An Intercomparison Exercise Using Irradiated Nickel Foils**

**R. C. Haight**

**Lawrence Livermore National Laboratory, Livermore, CA**

The International Atomic Energy Agency (IAEA) coordinates a number of nuclear-science research efforts under the title of Coordinated Research Programmes (CRP). One of these programs is devoted to the measurement and analysis of 14-MeV neutron nuclear data needed for fission and fusion reactor technology. A major activity of this CRP is the measurement of activation cross sections. To ensure reliable gamma-ray counting of activated samples, we undertook an intercomparison exercise at RTNS-II in which we irradiated a stack of 38 thin nickel foils of nearly equal source strength to yield 38 sources of  $^{57}\text{Co}$ ,  $^{58}\text{Co}$ , and  $^{60}\text{Co}$ . These foils were sent to the IAEA, and from there they were distributed to participating laboratories in approximately 16 countries. The participants will measure the radioactivity of the foils and send their results back to the IAEA for compilation and comparison. Once this analysis is completed, we will publish our results as an IAEA document.

## **Production of $^{74}\text{As}$ , $^{77}\text{Kr}$ , and $^{123}\text{Xe}$ by (n,2n) Reactions: Cross Sections and Decay Properties**

**J. Pawlikowski**

**U.S. Air Force**

**G. Coleman**

**Nebraska Wesleyan University, Lincoln, NE**

**C. F. Smith**

**Lawrence Livermore National Laboratory, Livermore, CA**

We irradiated sealed vials containing enriched  $^{78}\text{Kr}$ ,  $^{124}\text{Xe}$ , and pure  $^{75}\text{AsH}_3$  to produce  $^{77}\text{Kr}$ ,  $^{123}\text{Xe}$ , and  $^{74}\text{As}$  by (n,2n) reactions. By positioning the cells at angles between 30 and 90 deg to the beam, we were able to select neutron energies between 13.8 and 14.1 MeV for the determination of excitation functions. We used aluminum and nickel monitor foils to determine neutron fluence energy. We performed radioassay by gamma-ray spectrometry without removing the gases from the vials. In this way we could obtain decay data for both the gaseous (n,2n) products and their daughters. Four 1-h irradiations of five vials each were conducted.

The irradiations are as yet incomplete (four additional irradiations will be needed to provide the desired results). However, preliminary results have been published in an LLNL informal report (UCAR-10062). On completion of the experiments, the final results will appear in an LLNL formal report.



## **Production of $^{120m}\text{Sb}$**

**N. L. Smith**

**Nuclear Chemistry Division, Lawrence Livermore National Laboratory, Livermore, CA**

A supply of the radioactive isotope  $^{120m}\text{Sb}$  (half-life 5.8 days) was prepared by the reaction  $^{121}\text{Sb}(n,2n)^{120m}\text{Sb}$ .

The  $^{120m}\text{Sb}$  samples were then used to calibrate several of the Ge(Li) diode detectors operated by the Nuclear Chemistry Division.

There are no plans to publish this work.

## **Residence-Time Distribution of Oil Shale Particles in a Fluidized Bed**

**L. M. Lucht and D. Nelson**

**Lawrence Livermore National Laboratory, Livermore, CA**

We used scintillation detectors to measure the average time spent by particles of 12-70, 12-20, 20-40, and 40-70 mesh in regions of a 7-inch-diameter fluidized bed of crushed oil shale. Small samples of each particle-size range were irradiated for ten minutes by a pneumatic transfer system using the radionuclide  $^{29}\text{Al}$  (half-life 2.3 min). We found that both particle size and rate of gas flow through the bed affect residence-time distributions.

Related work was done previously by Mallon and Christiansen [1] using a smaller diameter fluidized bed. Consequently, wall effects were significant and the mass-flow rate was about half that found in the experiments just completed.

### **Reference**

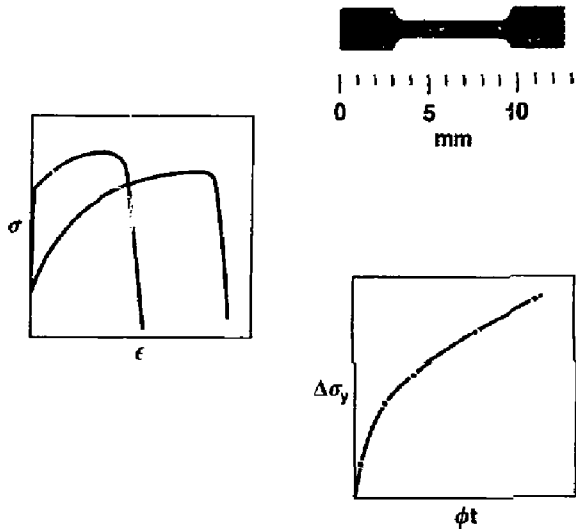
- [1] R. G. Mallon and D. E. Christiansen, "Median Residence and Dispersion Times for Fluidization of Crushed Oil Shale," Lawrence Livermore National Laboratory, Livermore, CA, UCRL-88492 (March, 1983). A version of this paper was also read at the *16th Oil Shale Symposium* held at Golden, CO (April, 1983).

## **14-MeV Neutron Sensitivity of Uranium Fission-Based Neutron Detectors**

**M. J. Moran and D. L. Redhead**

**Lawrence Livermore National Laboratory, Livermore, CA**

We have conducted a series of experiments at RTNS-II to measure the 14-MeV-neutron sensitivity of vacuum-diode neutron detectors. These detectors feature either a Uranium-235 or U-238 cathode. When neutrons impinge on the detector, the resulting fission events induce a signal that is proportional to the charge ejected from the cathode by fission fragments. The sensitivities obtained in these measurements were comparable to the canonical sensitivities for these detectors. The results also agreed with sensitivities inferred from measurements using the thermal-neutron flux at the Pennsylvania State University's reactor facility. This group of experiments is complete and there is no intention to publish the measurements.



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## Fusion-Neutron Irradiation of Multifilamentary Nb<sub>3</sub>Sn Alloy Superconductors

R. Flükiger

Kernforschungszentrum, Postfach 3640, D-7500 Karlsruhe, Federal Republic of Germany

M. W. Guinan

Nuclear Chemistry Division, Lawrence Livermore National Laboratory, Livermore, CA

The purpose of this experiment is twofold: first, we plan to investigate the possible effects of filament size on radiation degradation, and, secondly, we intend to compare the radiation response of unalloyed Nb<sub>3</sub>Sn with that of three other alloys.

The samples are all "bronze process" conductors. One of the two unalloyed samples contains 19 filaments and the other, 10,000. Three alloys are included in the experiment, two of which contain 19 filaments and are alloyed with 0.6% nickel and 1.6% titanium. The third contains 61 × 61 filaments and is alloyed with 7.0% tantalum.

Six samples of each conductor were packaged and mounted with respect to the target so that fluences of 3.2, 1.6, 0.8, 0.4, 0.2, and  $0.1 \times 10^{18}$  n/cm<sup>2</sup> would be delivered at the end of the irradiation. The experiment is combined with those of Cannon, Snead, and Guinan (see p. of this annual report).

At the end of 1984, about one-third of the desired fluence had been attained. We expect to complete the irradiation in March or April, 1985. After a one-month cooling period, the critical temperature (T<sub>c</sub>) of all 30 samples will be measured at LLNL and the samples will be shipped to Karlsruhe for critical current density (j<sub>c</sub>) measurements as a function of both strain and field. Dr. Flükiger will use the Grenoble facilities to carry out j<sub>c</sub> measurements to 24 T.

# Neutron Irradiation of Superconductors and the Damage-Energy Concept for Scaling Neutron Spectra

P. A. Hahn and H. W. Weber

Atominstytut der Österreichischer Universitäten, A-1070 Wien, Austria

M. W. Guinan

Nuclear Chemistry Division, Lawrence Livermore National Laboratory, Livermore, CA

L. R. Greenwood, R. C. Birtcher, and B. S. Brown

Materials Science Division, Argonne National Laboratory, Argonne, IL

Various stabilized single-core and multifilamentary NbTi superconductors, including the Swiss Large Coil Test (LCT) conductor, have been irradiated at room temperature at the RTNS-II in three runs up to a total fluence of  $5.1 \times 10^{17}$  n/cm<sup>2</sup> of monoenergetic 14.5-MeV neutrons. After each irradiation, we determined the critical current density ( $j_c$ ) in the range between 1.0 and 8.0 T. This determination was accomplished at 4.2 K with a standard four-probe technique and a 50- $\mu$ V/cm voltage criterion.

Identical sets of these materials have been irradiated under similar conditions with a spallation neutron spectrum of the Intense Pulsed Neutron Source (IPNS) in Argonne up to a fluence of  $1.9 \times 10^{18}$  n/cm<sup>2</sup> ( $E_n > 0.1$  MeV) and in the TRIGA-Mark II reactor in Vienna [1].

After a careful source characterization of these three irradiation facilities, the damage-energy cross section,  $E_d$ , was calculated for four different types of NbTi alloy (42, 46, 49, and 54 wt% titanium) [2, 3]. The radiation-induced degradation of  $j_c$  is found to scale (within experimental uncertainties) with the appropriate damage-energy cross sections multiplied by the fluence. As an example, Fig. 1 plots the fractional change of  $j_c$  of two commercial multifilamentary conductors versus the average damage energy displaced per atom.

This first explicit proof of the damage-energy concept for  $j_c$  variations in superconductors is considered to be most useful for the evaluation of radiation damage in superconductors under fusion reactor conditions.

The influence of the neutron spectrum in modifying the various flux pinning mechanisms operating in these materials has been analyzed in a Ph. D. dissertation by Peter Hahn [4]; an abstract of this study has been submitted to the *International Cryogenic Materials Conference*, to be held in Boston in 1985.

## References

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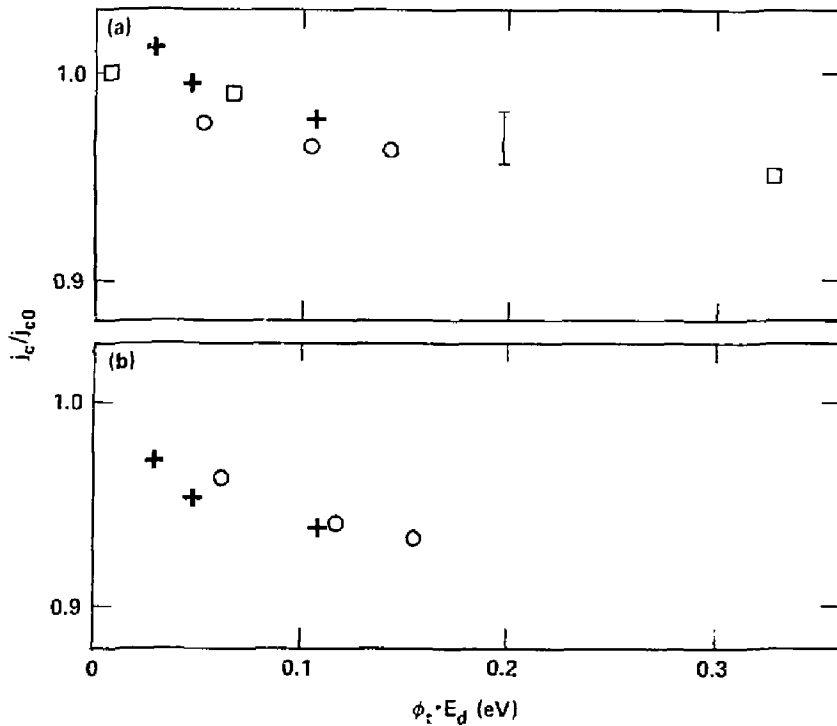


Figure 1. Fractional change of  $j_c$  at 5.0 T as a function of the average damage energy displaced per atom ( $\phi_t \times E_d$ ) for two NbTi multifilamentary conductors: (a) Nb, 49 wt% Ti, and (b) the Swiss LCT conductor (Nb, 46.5 wt% Ti). A representative error bar is shown in the upper data set (a).

- + = irradiation with spallation neutrons at IPNS,
- o = irradiation with 14.5-MeV neutrons at RTNS-II,
- = irradiation in the TRIGA-Mark II reactor, Vienna.