1982 Annual Report

Irradiations at the Rotating Target Neutron Source-II

Joint U.S.-Japan Sponsorship

The Cover:

Damage induced by 14-MeV neutrons on different materials. (Original photographs are courtesy of Professor M. Kiritani, Hokkaido University, Japan.)

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

MASTER

Lawrence Livermore National Laboratory

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

FOREWORD

As a result of discussions at the April 1983 meeting of the RTNS-II Steering Committee, it was agreed to publish an annual report. This publication would summarize irradiations and operations at RTNS-II.

This is the first annual report; it covers the period from the beginning of the Joint U.S.-Japan project until December 31, 1982. Compiled by D. G. Doran, the U.S. Point of Contact, under the direction of M. M. Cohen, OFE/DOE, this document discusses facility development and both fusion-related and non-fusion-related experiments. All irradiations are included. Many are secondary (or "add-on") in that they occupied a low flux location that did not affect the primary experiment.

Each summary article has been submitted by the investigator and has been altered only to meet the style and format restrictions of this report.

Clint Logan
Manager, RTNS-II

TABLE OF CONTENTS

	<u>Page</u>
Foreword	ii
<u>Facility</u>	
Introduction	1
(C. M. Logan)	
Facility Overview	3
(C. M. Logan)	
Operations	3
Physical Plant	3
Development	6
Other Items	7
Neutron Monitor Calibration	8
(D. W. Heikkinen)	
Effect of 14-MeV Neutron Irradiation on Thread-Locking Compound	9
(B. J. Schumacher)	
<u>Japanese-Initiated Experiments</u>	
Brief Review of Japanese Research Under U.S.-Japan Collaboration on RTNS-II Utilization	11
(K. Sumita)	
Radiation Damage of Electronic, Electric, and Optical Elements for Fusion Experiments	13
(T. Iida and K. Sumita)	
Induced-Activity Measurements for Fusion Materials (Al-Alloys and Stainless Steel)	19
(Y. Ogawa and T. Iida)	
Irradiation of Lithium Oxide/Titanium Carbide	21
(H. Matsui, T. Iida, and M. W. Guinan)	
Radiation Damage Process Analysis on Metallic Materials for Fusion	24
(M. Kiritani)	

U.S.-Initiated Experiments

Fusion Neutron Effects on Magnetoresistivity of Copper Stabilizer Materials	35
(M. W. Guinan and R. A. Van Konynenburg)	
Resistivity Damage Rate in Zirconium at 4.2 K	37
(M. W. Guinan and R. H. Zee)	
Resistivity Damage Rate of Nickel and Platinum at 4.2 K	39
(M. W. Guinan, R. A. Van Konynenburg, and J. H. Kinney)	
Effect of 14-MeV Neutron Irradiation on Loss Tangent of Aluminum Oxide	41
(J. D. Fowler, Jr. and F. W. Clinard, Jr.)	
Effect of 14-MeV Neutron Irradiation on MACOR Glass-Ceramic	43
(J. D. Fowler, Jr. and F. W. Clinard, Jr.)	
RTNS-II Irradiation (14-MeV Neutron) of Ceramic-Insulated Instrument Components in Support of FMIT Test-End Design	45
(C. P. Cannon)	
14-MeV Radiation Studies	47
(W. L. Barmore)	

U.S. Secondary Experiments

Antireflectivity Using Etched Nuclear Tracks	49
(R. W. Hopper)	
Phase-Matched Second-Harmonic Generation in Potassium Dihydrogen Phosphate After Neutron Bombardment	52
(M. A. Summers and M. S. Singh)	
Median Residence and Dispersion Times for Fluidization of Crushed Oil Shale	53
(R. G. Mallon)	
Preparation of ^{74}As	54
(H. G. Hicks)	
Isotopic Fractionation in Meteorites	55
(R. J. Borg)	

Effects of 14-MeV Neutron Bombardment on the Electrical Properties of Small-Geometry Semiconductor Devices and Integrated Circuits	57
(R. A. Hartmann and J. R. Srouf)	

The Nature of 14-MeV Neutron Damage in Silicon	58
(G. Woolhouse)	

Foreign

14-MeV Neutron Damage in Vanadium and Nickel	59
(D. Kaletta)	

Production of ^{93m}Nb Activity for Instrument Calibration	60
(G. Winkler and H. Vonach)	

FACILITY

INTRODUCTION

(C. M. Logan)

Early in 1982, a unique collaboration began between the Office of Fusion Energy (OFE) of the U.S. Department of Energy (DOE) and Monbusho, the Japanese Ministry of Education, Science, and Culture. At that time the two countries agreed to operate, as equal partners for five years, the Rotating Target Neutron Source (RTNS-II) at Lawrence Livermore National Laboratory (LLNL), Livermore, California.

The RTNS-II is a deuterium-tritium neutron source producing the world's most intense beam of 14-MeV neutrons. It is dedicated to the study of problems concerning the development and selection of materials for fusion reactors.

The RTNS-II program has three primary objectives. One is the direct testing of a material to be used in a fusion reactor component. Lifetime exposures are possible in some cases, e.g., superconducting magnet components. A second objective is to develop quantitative information to aid in correlating 14-MeV and fission neutron radiation effects. A third objective is to learn more about damage production by high energy neutrons.

Although many of the initial experiments were exploratory, we made substantial progress toward these objectives during this reporting period. Specifically, we gained important new information on the behavior of electrical, electronic, and optical components under 14-MeV neutron irradiation; especially pertinent are the new measurements of the magneto-resistivity of copper used as a stabilizer in superconducting magnets.

Collaboration between the two countries on irradiation experiments is an important aspect of this program. During 1982, we shared irradiation space in a cryogenic experiment, and made plans for sharing space in an elevated

*Work performed under the auspices of the U.S. Department of Energy and Japan Monbusho by Lawrence Livermore National Laboratory under contract number W-7405-ENG-48.

temperature experiment scheduled for early in 1983. More significantly we are organizing cryogenic experiments on superconductors and organic insulators as well as an in-situ study of volume changes in inorganic insulators.

We also improved the RTNS-II facility this year. Our primary objective was to increase the production rate of 14-MeV neutrons through development of a dependable large (50-cm-diam) target. We anticipate that a reliable, continuous supply will be available by early 1983. Upgrading of power supplies has resulted in routinely achieving 100 mA of beam on target. Work has begun on bringing the second ("right") neutron source into operation.

Finally, offices for staff, especially visiting Japanese scientists, and facilities for limited post-irradiation testing were completed. The latter features a 200-kV transmission electron microscope (TEM) and a x-ray diffractometer, both provided by the Japanese. Also included is a scanning electron microscope (SEM).

FACILITY OVERVIEW

C. M. Logan

Operations

The RTNS-11 facility contains two independent 14-MeV neutron sources. Only one, the "left" machine, was in operation during this reporting period. Nominal operation was two shifts per day, five days per week.

Each source is an air-insulated Cockroft-Walton accelerator producing a 400-keV deuteron beam that strikes a titanium-titride target. The coated copper-alloy target is water-cooled and rotated at nominally 5000 rpm (Fig. 1). Both 23-cm- and 50-cm-diam targets were used during this report period, although the latter will soon be used exclusively. The neutron source, i.e., beam diameter, is about 1 cm; hence, the neutron flux decreases rapidly and nearly isotropically with distance.

During this year we conducted elevated temperature experiments using the Hanford Engineering Development Laboratory (HEDL) two-temperature zone furnace. Figure 2 shows this device just prior to final positioning to within approximately 0.5 mm of the target assembly. The furnace maintains samples in a vacuum at two elevated temperatures (usually 80^o and 300^oC) during irradiation.

Continuous operation for selected experiments began in December 1982. Initial operation of the right machine is now planned for May 1983. Full 15-shift per week operation of the left machine will begin in July 1983.

Physical Plant

We moved into our new office building on December 20, 1982. An additional structure has been added to the facility for storage of non-radioactive supplies and components.

Japanese equipment used for post-irradiation testing has been installed in the materials test laboratory. The SEM device, previously in the hot work room at RTNS-11, was moved to this lab. A dark room was installed nearby.

Figure 1. Schematic of a section of the Target System. The target rotates at 5000 rpm. The angle between the axis of rotation and the deuteron beam is slowly varied to use the entire tritium-coated band on the inside of the target. The neutron source remains stationary in the room. To acquire maximum neutron flux, samples are placed as close as possible to the exterior surface of the spinning target.

Figure 2. Elevated temperature irradiations at RTNS-II using the HEDL two-temperature zone furnace.

Development

Both design review and initiation of orders for spare parts were completed during the summer of 1982. Consultation with the manufacturer of our high-voltage equipment led to modest design changes to make the diode string more resistant to damage during spark-down.

The acceleration column design was reviewed in June. We decided to make substantial changes in the original RTNS-II design. These changes eliminate ceramic-to-metal brazing, improve electrostatic design, and increase shielding to reduce beam-related radiation reaching the insulators. The beam optics of the new column are unchanged. This design is unconventional, and there is some risk of poor performance. Because of this, we are making a single prototype column at this time and plan to begin right machine operation with the original design.

To achieve low x-ray fluxes at the acceleration column ceramics, it is important to prevent electrons from "backstreaming" through regions of high voltage gradient. New ground electrodes are fabricated that have improved vacuum conductance and electron suppression.

Development work on the right machine during the summer convinced us that upgraded power supplies for the ion-source arc and for ion-source extraction were essential for increasing beam. In September and October, we replaced ion-source-arc power supplies on both machines. With this improvement, we are capable of routinely delivering greater than 100 mA on target. An order has been placed for higher current and voltage extraction supplies. We expect to install these during late summer of 1983.

We estimate that the left machine will again be converted for use of 50-cm-diam targets by April or May of 1983. Our large target development program is progressing as planned; we anticipate a reliable, continuous supply by April 1983.

In addition, we have begun a joint program with Professor Tadashi Yanagi of Osaka University to explore methods of improving the coatings used to contain tritium on RTNS-II targets. Initially, we will investigate the performance of sputter-deposited titanium compared to the conventional

evaporated coatings now in use. Later, work may involve alternate coating materials. We are also considering joint work in other tritium technology areas including methods of decontamination.

We need improved methods of measuring position and size of both the beam and the neutron source itself. We have made substantial progress but much work remains.

The primary method of target manufacture utilizes a thick (approximately 0.5 mm), electroplated, copper layer. A disadvantage of this method is that the electrodeposited copper has low mechanical strength after heating during processing. We have started a program to develop a higher strength, copper-based, electroplated material that will still retain high conductivity. Two methods are being explored: alloying and codeposition of ceramic particles.

Beginning in October 1983, targets will be coated with titanium and loaded with tritium at LLNL in a system that is now being constructed. We expect that this will result in better, more consistent targets, shorten lead time, and eliminate the problems inherent in transportation of radioactive material.

Other Items

The Guide for Experimenters was reissued in accordance with the instructions of the April 1982 meeting of the RTNS-11 Steering Committee.

TITLE:

Neutron Monitor Calibration

INVESTIGATOR:

D. W. Heikkinen

AFFILIATION:

RTNS-II Facility, Lawrence Livermore National Laboratory

SPONSORING PROGRAM:

DOE and Monbusho

SUMMARY:

Neutron production at RTNS-II is monitored using ionization chambers and proton recoil counters. These monitors are used to provide both instantaneous and cumulative results for neutron production and the time-history of any irradiation.

As an ongoing program, these monitors are periodically calibrated using niobium dosimetry foils at each monitor location. Using these calibrations, neutron production results can be given with an accuracy of 3 to 5%.

PUBLICATION STATUS:

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

TITLE:

Effect of 14-MeV Neutron Irradiation on Thread-Locking Compound

INVESTIGATOR:

B. J. Schumacher

AFFILIATION:

RTNS-11 Facility, Lawrence Livermore National Laboratory

SPONSORING PROGRAM:

OFE/Materials and Radiation Effects Branch

SUMMARY:

Proper functioning of the RTNS-11 rotating target assembly depends on Loctite #242. Throughout the assembly, threads are treated with the compound to make them resistant to loosening under extremes of load cycling, thermal cycling, and vibration. Response of this material to radiation is unknown. We prepared an add-on experiment to measure the response to 14-MeV neutrons. Post-irradiation measurements were made of torque required for breakaway and continuous turning after breakaway.

The results lack plausibility. We suspect inconsistent curing conditions. The experiment will be modified and redone.

PUBLICATION STATUS:

No publication is planned at this time.

JAPANESE-INITIATED
EXPERIMENTS

TITLE:

Brief Review of Japanese Research Under U.S.-Japan Collaboration on
RTNS-II Utilization

INVESTIGATOR:

K. Sumita

AFFILIATION:

Japanese Point of Contact

SUMMARY:

In the middle of February 1982, the first Japanese experiment using RTNS-II was started. Since then, some Japanese samples have always been under irradiation. Though the preparation time for the planned experiments was very restricted, experiments were carried out successfully with the extensive cooperation of U.S. colleagues at Lawrence Livermore National Laboratory.

Major Japanese research fields selected for 1982 were the following:

(i) Radiation Damage on Electronic, Electric, and Optical Elements for Fusion Experiments; (ii) Induced-Activity Measurements for Fusion Materials; (iii) Irradiation of Lithium Oxide and Titanium Carbide at RTNS-II; and (iv) Radiation Damage Process Analysis on Metallic Materials for Fusion. Of course, some experiments in other categories were introduced as "add-on" experiments from the fusion research field.

Five researchers, Kenji Sumita, Toshiyuki Iida (Osaka Univ.), Hisayuki Matsui (Nagoya Univ.), Michio Kiritani (Hokkaido Univ.), and Chihiro Ichihara (Kyoto Univ.) were sent from Japan to the RTNS-II site as resident exchange scientists during this period. Over 60 researchers, mainly from Japanese national universities and some from government-controlled research organizations, have made their contributions and their support in various ways to the research of the above-mentioned scientists sent from Japan. Some test-materials preparation, and pre- and post-irradiation examination services were also supplied through the courtesy of several research organizations and companies.

PUBLICATION STATUS:

This report is a very brief review of those fruitful research results that were obtained by the end of 1982. Full reports will be published as an Annual Review Report of Japanese Participations for U.S.-Japan Collaboration on RTNS-II Utilization 1982.

TITLE:

Radiation Damage of Electronic, Electric, and Optical Elements for Fusion Experiments

INVESTIGATORS:

T. Iida and K. Sumita

AFFILIATION:

Osaka University

SUMMARY:

A. Optical Fiber and Optoelectronic Device Irradiation Test*

Three kinds of commercially available optical fibers and three typical optoelectronic devices (light-emitting diode or LED, a PIN silicon photodiode, and photomultiplier) were irradiated with 14-MeV neutrons at the RTNS-II facility. The in-situ irradiation test was made at room temperature.

It was ascertained from the irradiation tests that the Ge-doped silica core fiber suffered from a high optical absorption loss because of the 14-MeV neutron irradiation; on the other hand, induced losses of the pure silica core fibers were not so large and also recovered quickly. Moreover, it was found that the pure silica core fiber with a high (OH) content had a higher absorption loss soon after irradiation than the other fiber, but that its induced loss had a tendency to saturate with increasing neutron fluence. This means that the pure silica core fiber with a high (OH) content is more suitable for use in a high neutron fluence field.

*Some samples and domestic experiments were courtesy of Japanese manufacturing companies.

The quantum efficiency of the optoelectronic devices all decreased with increasing neutron fluences. The LED and the PIN silicon photodiode, made from semiconductor materials, were especially sensitive to 14-MeV neutrons and influenced even in the low fluence region below 10^{11} n/cm². The PIN silicon photodiode also suffered from the degradation of the response time to a light pulse input. The photomultiplier experienced a decline in the gain beyond 2×10^{14} n/cm² and a rise in the background noise level. As regards the decline of the gain of the photomultiplier, it is not clear from this irradiation test which part of the photomultiplier--the photocathode, electrodes such as dynodes and an anode, a quartz window, or others--suffered from the damage.

The results of the irradiation tests for the optical fibers and the optoelectronic devices are summarized in Fig. 3.

B. Electronic Device Irradiation Tests

The irradiation tests for semiconductor electronic devices were made at room temperature. Many samples, more than thirty for each model of the devices, were set around the target. The irradiated samples were examined concerning the static characteristics, mainly the gain, within one day after irradiation. Figure 4 summarizes the results of the irradiation tests for various semiconductor electronic devices.

C. Radiation Detector Irradiation Test

Five different commercially available radiation detectors were the first to be irradiated with 14-MeV neutrons at RTNS-11. These irradiation experiments were performed to determine how long the detectors can be used in the fusion neutron field. The detectors were silicon surface-barrier detectors (SSD), inorganic scintillators of ZnS and CsI, plastic scintillators, and liquid scintillators.

The multichannel spectrometry system was utilized for examination of the detectors. Spectral response of the detectors to standard radiation sources was measured before and after irradiation. If the detectors suffered from

Figure 3. Relative efficiency vs 14-MeV neutron fluence for various optical devices.

Figure 4. Relative gain vs 14-MeV neutron fluence for various electronic devices.

damage such as the degradation of the quantum efficiency after irradiation, the peaks of the standard sources shifted to the lower energy side. The positions of the peaks were compared. Figure 5 summarizes the results.

D. Effects of 14-MeV Neutron Irradiation on Polymer Composites*

Ten polymer composites were irradiated with 14-MeV neutrons at room temperature to neutron fluences of 1.9×10^{16} to 5.0×10^{17} n/cm². The polymer composites were composed of reinforcements and matrix resins. Mechanical and electrical properties were measured before and after irradiation. Color and transparency showed considerable changes, but there was no significant degradation of compressive strength and insulation resistance. None of the composites suffered significant damage by a ⁶⁰Co gamma-ray dose to 1×10^9 rads.

PUBLICATION STATUS:

D-T Neutron Irradiation Experiments on Optical Fibers by RTNS-II

T. Iida, K. Sumita (Osaka U.)

B. J. Schumacher and G. M. Logan (LLNL)

A 46, Fall Meeting of the Atomic Energy Society of Japan, October 1982

D-T Neutron Irradiation Experiments on Organic Insulators by RTNS-II

T. Iida, K. Sumita (Osaka U.)

K. Kadotani, I. Sone (Hitachi)

N. Terazawa and T. Inohara (Toshiba)

B 30, Annual Meeting of the Atomic Energy Society of Japan, March 1983

D-T Neutron Irradiation Experiments on Radiation Detecting Systems and Their Components by RTNS-II

T. Iida and K. Sumita (Osaka U.)

B 31, (ibid.)

*Some samples and domestic experiments were courtesy of Japanese manufacturing companies.

Figure 5. Relative efficiency vs 14-MeV neutron fluence for various radiation detectors.

TITLE:

Induced-Activity Measurements for Fusion Materials (Al-Alloys and
Strainless Steel)

INVESTIGATORS:

Y. Ogawa and T. Iida

AFFILIATIONS:

Nagoya University and Osaka University

SUMMARY:

We have paid considerable attention to reducing the induced activity of structural materials. From this point of view, Al-alloys are being considered for use by the tokamak of burning plasma study at I.P.P., although the conventional tokamak is made of stainless steel. The calculation was performed with the THIDA code, which was developed by JAERI. To check our calculation results and estimate the induced activity due to impurities in commercially available Al-alloy, the following were irradiated:

Case A: (i) Al-2017 (ii) Al-6063 (iii) Al-7075

Case B: (iv) SUS 304 (v) high Mn steel

In Fig. 6 we compare typical experimental results for gamma rays from Al-6063 after two months with the results calculated by THIDA code.

PUBLICATION STATUS:

Induced Activity Experiments of Fusion Materials by RTNS-II

Y. Ogawa (I.P.P., Nagoya U.) and T. Iida (Osaka U.)

B 32, Annual Meeting of the Atomic Energy Society of Japan, March 1983.

Total neutron fluence = 6.6×10^{16} n

Weight of Al-6063 = 1.36 g

Time after irradiation = 2 months

Hg: 0.51% Fe: 0.16% Ti: 40 ppm Zn: 30 ppm

Si: 0.38% Cu: 60 ppm Mn: 30 ppm Ni: <10 ppm
(4 ppm)

Zr: <5 ppm (Nb: 10 ppm Mo: 30 ppm)

Figure 6. Comparison between experimental and calculational data for Al-6063.

TITLE:

Irradiation of Lithium Oxide and Titanium Carbide*

INVESTIGATORS:

H. Matsui, T. Iida, and M. W. Guinan

AFFILIATIONS:

Nagoya University, Osaka University, LLNL

SUMMARY:

In the present work, single crystals of lithium oxide (Li_2O) were irradiated at room temperature in a dose range between 2.34×10^{16} and $2.28 \times 10^{17} \text{ n/cm}^2$. After that, the lattice parameter was examined to study the stability of crystal structure under fast neutron irradiation. On the other hand, for three kinds of titanium carbide (TiC), we carried out in-situ electrical resistivity measurements at liquid helium temperatures at a dose of $2 \times 10^{17} \text{ n/cm}^2$ with successive isochronal annealings up to 340 K to discern the recovery from irradiation-induced defects.

A. Li_2O

For the single crystal Li_2O used here, the lattice parameter (a_0) was 0.46120 nm before irradiation. At an irradiation of $2.34 \times 10^{16} \text{ n/cm}^2$ (the lowest irradiation dose in this work), a slight expansion was already revealed in the crystal lattice. A curious change of the lattice parameter was found with increasing neutron fluence, as indicated in Fig. 7. A reduction of the lattice expansion was observed at around 10^{17} n/cm^2 and, with further irradiation, the lattice parameter expanded again. The same trend was observed in change of the line broadening of the diffraction profile, though the change was quite small.

*Single crystals of Li_2O were obtained through the courtesy of JAERI.

Figure 7. Change of lattice parameter in Li_2O as a function of neutron fluence.

The lattice expansion ($\Delta a/a = 1 \times 10^{-3}$) of polycrystalline Li_2O has been reported in an irradiation by fission neutrons (using JRR-2) to $1 \times 10^{17} \text{ n/cm}^2$ (thermal neutron fluence). It is supposed that the expansion above 10^{17} n/cm^2 in the present case might be attributed to an accumulation of irradiation-induced vacancies. At the present stage, however, it is not easy to explain the strange behavior of the lattice parameter of irradiated Li_2O obtained here. This could be clearly done by direct observation of the induced defects by a transmission electron microscope.

B. TiC_x

To examine the effect of nonstoichiometry on radiation damage, we irradiated three kinds of TiC_x with different carbon contents. Increases of the electrical resistivity were found in each sample, and straight inclination was observed in the plots of the resistivity vs neutron fluence. Damage rate, as defined as $d(\Delta\rho)/d\phi$, differed in each sample. The damage rate appeared to depend on the nonstoichiometry: the higher the carbon content, the higher the damage rate.

After the in-situ resistivity measurements, the specimens were isochronally annealed to 340 K. Precise measurements, however, were performed from 33 K. A big recovery stage was revealed between 140 and 240 K with two small stages around 100 and 280 K. Those low-temperature recovery stages might be attributed to movements of the interstitial-type defects. Vacancy-type defects must be annealed at higher temperatures. A large portion of the induced resistivity still remained even after the 340 K anneal in both the low temperature fast-neutron irradiation as well as in the fission-neutron-irradiated TiC_x .

PUBLICATION STATUS:

Irradiation Experiments at RTNS-II (Li_2O , TiC)

H. Matsui (Nagoya U.) et al.

Spring Meeting of Japan Society of Metals, April 1983.

TITLE:

Radiation Damage Process Analysis on Metallic Materials for Fusion

INVESTIGATOR:

M. Kiritani

AFFILIATION:

Hokkaido University

SUMMARY:

The experimental research plan was made on the understanding that RTNS-II is not strong enough to test the materials up to the stage of the practical fusion environment but is strong enough to acquire fundamental understanding of the characteristic of radiation damage by fusion neutrons. Another factor which has been strongly considered in the planning was to effectively utilize this first experimental run to make all the related Japanese scientists acquainted with this experimental method for the purpose of prospective further experiments. The principal method adopted is the observation of microstructure change by neutron irradiation with transmission electron microscopy, and the present principal investigator has been appointed as one of the most experienced scientists both in the related field of research and in the experimental techniques.

Names and affiliations of the participants are as follows:

M. Kiritani, Hokkaido University	H. Yoshida, Kyoto University
T. Takeyama, Hokkaido University	Y. Shimomura, Hiroshima University
H. Takahashi, Hokkaido University	K. Kitajima, Kyushu University
S. Ishino, University of Tokyo	N. Yoshida, Kyushu University
H. Kawanishi, University of Tokyo	E. Kuramoto, Kyushu University
A. Kohyama, University of Tokyo	C. Kinoshita, Kyushu University

Materials and the treatment of specimens prepared for the irradiation are listed in Table 1. Objectives of each given treatment are multipurpose; examples are:

1. Detection of free defects from the change of pre-existing defects
2. Influence on cascade stability by pre-existing defects
3. Effect on cascade stability by minor elements
4. Application of order diffraction observation
5. Detection of the difference in various metallurgical phases
6. Variation of cascade stability with characteristic temperature of point defects

Almost half of the total 200 loaded samples had been pre-thinned for electron microscope observation, and two-thirds of the pre-thinned samples had been loaded between thin aluminum half-disks to minimize the mass of the materials (less than 0.005 mg for each). Numbers of samples, especially those which were given specific treatments, had been observed before irradiation, and their characteristic structures were recorded.

To prevent the contamination and oxidation of samples after their preparation until the end of the irradiation before observation, a compact vacuum-sealed chamber was designed and constructed. The irradiation started on July 13 and continued for 3 weeks (every day 15 hours, 5 days a week) using a newly installed 50-cm-diam target to reach 1×10^{18} n/cm² at the front chamber surface.

A high resolution electron microscope Model JEM-200CX was installed in the RTNS-II Laboratory in the early part of July 1982, and it proved its required performance of 1.4-Å resolution.

Examinations of about 40 specimens, which could not be handled as nonradioactive materials because their induced radioactivity would not cool down within one month, were performed at the RTNS-II Laboratory. More than 1000 TEM photographs of the irradiation results will be analyzed by the research participants; about 50 specimens in which the radioactivity was originally very weak or had cooled down sufficiently are now under observation in Japan.

Table 1. Materials and treatments of samples irradiated.

	Material	Treatment
1	Al	Annealed
2	Al	Cold worked
3	Al	Doped with V-dislocation loops by quenching
4	Al	Doped with voids by quenching
5	Al	Doped with I-dislocation loops by electron irradiation
6	Al-dilute alloys	Ag, Mg & Si up to 500 at.ppm
7	Al-Cu	With Gr. zones
8	Au	Annealed
9	Au	Doped with stacking fault tetrahedra by quenching
10	Cu	Annealed
11	Fe	Cold worked
12	Fe	Pre-irradiated with electrons
13	Fe-C	Solution treated
14	Ge	Single crystal
15	MgO	As grown
16	Si	Purified
17	Ni	Annealed
18	Ni	Cold worked
19	Ni	Doped with I-dislocation loops by electron irradiation
20	Ni	Doped with voids and bubbles by ion irradiation
21	Ni	He ion pre-irradiated
22	Mo	Cold worked
23	Mo	Pre-irradiated with electrons
24	V	Cold worked
25	V-C	Solution treated
26	V-C	c-phase precipitated
27	Cu ₃ Au	Ordered
28	Ni ₃ Al	Ordered
29	NiAl	Ordered
30	FeAl	Ordered
31	TiNi	Ordered
32	TiC	Near stoichiometry
33	Fe-Cr	Various compositions with and without C
34	Fe-Cr-Ni	" " "
35	Fe-Cr-Mo	" " "
36	Fe-Cr-Nb	" " "
37	Fe-Cr-Ti	" " "
38	Fe-Cr-Ni-Ti	" " "
39	316 SS	Variously modified
40	Ferritic steels	Ferrite only
41	Ferritic steels	Ferrite and martensites
42	Amorphous FeB	14, 20 and 25 at.% B
43	Polymers	3 kinds
44	SiO ₂	For optical measurements

Data acquisition and analysis have not been completed, and only a brief quotation of topics is given with some representative photographs.

- a. Density and Size of Survived Defects--Volume number density of survived defects has been obtained by the thickness gradient method or stereoscopic observation. Cross sections of sample neutron collisions that survived defects are 1.3, 3.9, 1.2, and 2.6 barns for Au, Ni, Cu₃Au, and Ge, respectively. Size distributions of these samples are now under analysis using PKA energy distributions.
- b. Sub-Cascade Damage--Defects formed from sub-cascade damage have been most clearly observed in gold as in Fig. 8. The distribution of the size of the sub-cascade groups counted by the number of sub-cascades contained in a group is shown in Fig. 9. This figure indicates that the majority of defects belongs to the sub-cascade damage groups.
- c. Influence of Dislocations on the Survival of Defects--Figure 10 shows a typical case of cold-worked molybdenum in which a pronounced enhancement of the formation of dotted defects is observed on or in the vicinity of the majority of pre-existing dislocations. This effect has been observed in many other materials.
- d. Voids and Bubbles--The influence of the existence of cavities could be detected by irradiating specimens in which cavities had been already introduced prior to the neutron irradiation. Figure 11 shows the case of Ar⁺ ion-irradiated nickel in which a remarkable enhancement of the formation of defects is observed around each cavity.
- e. Disordered Zones--In addition to the observation of disordered zones in ordered alloys, unstable disordered zones have been detected in semiconductor germanium in which the neutron-irradiation-induced defect structures disappear during the observation with 200-keV electrons as illustrated in Fig. 12.

Figure 8. (a) Numbers of sub-cascade damage in gold.
 (b) Distribution of individual defects in cascade groups.

Figure 9. Defects clearly showing sub-cascade damage in gold: Au (B-23), 3.9×10^{17} n/cm².

Figure 10. Influence of dislocations on the formation of defects from cascade damage: The case of deformed Mo (C-9), $5.9 \times 10^{17} \text{ n/cm}^2$.

Figure 11. More numbers of secondary defect formation observed in the vicinity of the cavity in Ni (K-73), 4.7×10^{17} n/cm². The cavities had been introduced by Ar⁺ ion irradiation at 600°C prior to the neutron irradiation.

Figure 12. Gradual annihilation of defects formed in Ge (K-83) during observation with 200-keV electrons. Neutron irradiation was up to $4.1 \times 10^{17} \text{ n/cm}^2$.

PUBLICATION STATUS:

Research Project of 14-MeV Neutron Irradiation of Materials with RTNS-II

M. Kiritani

Annual Meeting of the Japan Institute for Metals, September 1982.

D-T Neutron Irradiation Damage of Materials with RTNS-II

I. Report on the First Irradiation Experiment of Materials

M. Kiritani

B 33, Annual Meeting of the Atomic Energy Society of Japan, March 1983.

D-T Neutron Irradiation Damage of Materials with RTNS-II

II. Brief Review of Observed Results

M. Kiritani

P 1, Annual Meeting of the Atomic Energy Society of Japan, March 1983.

14-MeV Neutron Irradiation (RTNS-II) Damage of Gold

M. Kiritani, Y. Shimomura, K. Kitagawa, and N. Yoshida,

Spring Meeting of the Japan Society for Metals, April 1983.

14-MeV Neutron Irradiation (RTNS-II) Damage of Aluminum

Y. Shimomura, K. Kitagawa, S. Ishino, K. Kawanishi, N. Yoshida,
and M. Kiritani

Spring Meeting of the Japan Society of Metals, April 1983.

14-MeV Neutron Irradiation (RTNS-II) Damage of Ordered Cu_3Au

M. Kiritani, N. Yoshida, S. Kojima, and M. Sugiyama

Spring Meeting of the Japan Society for Metals, April 1983.

14-MeV Neutron Irradiation (RTNS-II) Damage of V and V-C Alloys

T. Takeyama, H. Takahashi, S. Ohonuki, and M. Kiritani

Spring Meeting of the Japan Society of Metals, April 1983.

14-MeV Neutron Irradiation (RTNS-II) Damage of Germanium

M. Kiritani and M. Hirata

Spring Meeting of the Japan Society of Metals, April 1983.

U.S.-INITIATED EXPERIMENTS
**(Department of Energy/
Office of Fusion Energy)**

TITLE:

**Fusion Neutron Effects on Magnetoresistivity of Copper Stabilizer
Materials**

INVESTIGATORS:

M. W. Guinan and R. A. Van Konynenburg

AFFILIATION:

Lawrence Livermore National Laboratory

SPONSORING PROGRAM:

**Department of Energy, Office of Fusion Energy, Materials and Radiation
Effects Branch, Special Purpose Materials Task IV.G.2--Radiation Damage
in Superconducting Composites**

SUMMARY:

The objective of this work is to quantify the changes that occur in the magnetoresistivity of coppers (having various purities and pretreatments as well as at magnetic fields up to 12 T) during the course of sequential fusion neutron irradiations at about 4 K and anneals to room temperature. In conjunction with work in progress by Coltman and Klabunde of ORNL, the results should lead to engineering design data for the stabilizers of superconducting magnets in fusion reactors. These magnets are expected to be irradiated during reactor operation and warmed to room temperature periodically during maintenance.

Eight copper wires were repeatedly irradiated at 4.2 to 4.4 K with 14.8-MeV neutrons and isochronally annealed at temperatures up to 34°C for a total of five cycles. Their electrical resistances were monitored during irradiation under zero-applied magnetic field. After each irradiation, the magnetoresistances were measured in applied transverse magnetic fields of up to 12 T. Then the samples were isochronally annealed to observe the recovery of the resistivity and magnetoresistivity. After each anneal at the highest temperature (34°C), some of the damage remained and contributed to the damage state observed following the subsequent irradiation. In this way, we were able to observe how the changes in magnetoresistance would accumulate during the

repeated irradiations and anneals expected to be characteristic of fusion reactor magnets. For each succeeding irradiation, the fluence was chosen to produce approximately the same final magnetoresistance at 12 T, taking account of the accumulating residual radiation damage. The increment of magnetoresistivity added by the irradiation varied from 35 to 65% at 12 T and from 50 to 90% at 8 T for the various samples.

We found that a Kohler plot determined by varying only the magnetic field on a sample was adequate to describe the effects on the magnetoresistivity of subsequent variations in the zero-field resistivity brought about by cold work, irradiation, and annealing. This makes it possible to predict the behavior of stabilizer materials during their lifetime in a fusion reactor from measurements made before magnet fabrication.

We also found that the fraction of irradiation-produced zero-field resistivity which remained after successive irradiation-anneal cycles decreased as the total amount of damage resistivity produced at 4.2 K was increased. This held true up to the maximum accumulated 4.2-K damage resistivity studied, which was $120 \times 10^{-11} \Omega\text{m}$.

From consideration of these data and a preliminary design of a LHeII-cooled magnet system (1.8 K), it appears that the damage to the polyimide thermal insulation rather than the increase in magnetoresistivity of the stabilizer will be the determining factor for shielding requirements in systems of this type.

Upon the completion of planned experiments at the Intense Pulsed Neutron Source by Coltman and Klabunde of ORNL using the same sample materials as were used here, it will be possible, from all the data, to make accurate predictions of the magnetoresistive behavior of copper stabilizers under expected fusion reactor conditions. Accordingly, we do not plan to perform further 4-K copper irradiations. Future experiments will be concerned with superconductor materials and with copper above room temperature.

PUBLICATION STATUS:

This experiment and the analysis of data for three of the samples will be reported in the Fifth Annual Progress Report on Special Purpose Materials for Magnetically Confined Fusion Reactors. Analysis of the data for all eight samples will be reported at the Third Topical Meeting on Fusion Reactor Materials at Albuquerque, New Mexico, on September 19-22, 1983.

TITLE:

Resistivity Damage Rate in Zirconium at 4.2 K

INVESTIGATORS:

M. W. Guinan and R. H. Zee

AFFILIATION:

Lawrence Livermore National Laboratory/Chalk River Nuclear Laboratories

SPONSORING PROGRAM:

Department of Energy, Office of Fusion Energy, Materials Radiation
Effects Branch, Damage Analysis and Fundamental Studies Program/Materials
Science Branch, Chalk River Nuclear Laboratories

SUMMARY:

High-purity zone-refined zirconium wire, provided by R. H. Zee, was irradiated to a dose of 1.8×10^{17} n/cm² at 4.2 K. The resistance of the wire was measured during irradiation at dose increments of $\sim 1.2 \times 10^{15}$ n/cm², providing a detailed damage rate history. Following irradiation, isochronal annealing was performed up to 340 K. This irradiation was carried out simultaneously with an irradiation of TiC by H. Matsui of Nagoya University.

The results of the damage rate study reveal a pronounced concave curvature in a plot of $d\Delta\rho/d\phi$ vs $\Delta\rho$. The curvature persists over the whole range of $\Delta\rho$ investigated (up to 1100 nΩ-cm). Comparing our results to those from irradiations in a pure fission spectrum and a reactor spectrum on a damage energy basis, we find differences of $\pm 25\%$. This is a considerably larger scatter than for the results of studies on 8 cubic metals, which were all within $\pm 10\%$. While the additional scatter is in part due to the effect of the pronounced curvature of the damage production on extrapolation of $d\rho/d\phi$ to zero damage, the effect of differing sample purity may also play a role. The present results will later be compared to those from an irradiation on identical material at the intense pulsed neutron source (IPNS) at Argonne National Laboratory. This comparison will eliminate possible variations due to sample materials.

PUBLICATION STATUS:

The preliminary comparison with the results of fission neutron irradiations will appear in the Damage Analysis and Fundamental Studies Quarterly. A comparison, including the IPNS irradiation as well, will be submitted to the Third Topical Meeting on Fusion Reactor Materials at Albuquerque, New Mexico, September 19-22, 1983.

TITLE:

Resistivity Damage Rate of Nickel and Platinum at 4.2 K

INVESTIGATORS:

M. W. Guinan, R. A. Van Konynenburg, and J. H. Kinney

AFFILIATION:

Lawrence Livermore National Laboratory

SPONSORING PROGRAM:

Department of Energy, Office of Fusion Energy, Materials and Radiation Effects Branch, Damage Analysis and Fundamental Studies Program (DAFS)

SUMMARY:

Platinum and nickel foils of nominal purity (99.95% to 99.99%) were irradiated to a fluence of 1.8×10^{17} n/cm² at 4.2 K. The resistances of the samples were monitored at fluence increments of 1.2×10^{15} n/cm² during irradiation. Following irradiation, isochronal annealing was performed up to 340 K. The irradiation of these two samples completes our study of fission-fusion correlations of defect production in cubic metals.

Vanadium, niobium, molybdenum, tungsten, aluminum, and copper had previously been irradiated at RTNS-I or RTNS-II and the results compared to those obtained in fission neutron spectra.

Initial resistivity damage rates in both Ni and Pt can be predicted to within 10% by damage energy scaling from the results of irradiations in a pure fission spectrum. Damage energy scaling to $\pm 10\%$ was also observed for the six metals previously irradiated. From the results on all eight cubic metals, we conclude that total defect production, as measured by resistivity changes at 4.2 K, can be predicted in fusion environments from the results of neutron irradiation studies in fission spectra to $\pm 10\%$. This experiment was carried out simultaneously with the 4.2-K irradiation at TiC by H. Matsui of Nagoya University.

PUBLICATION STATUS:

- (1) Initial Damage Rates: Results for V, Nb, Mo, W, Al, and Cu were published in "Resistivity Damage Rates in Fusion-Neutron Irradiated Metals at 4.2 K," by M. W. Guinan and J. H. Kinney, J. Nucl. Mater. 108 and 109, 95 (1982). The results for Pt and Ni will appear first in the DAFS Quarterly, and subsequently at the Third Topical Meeting on Fusion Reactor Materials in Albuquerque, NM on September 19-22, 1983.
- (2) Annealing Studies: A comparison of the results of annealing studies in all four fcc metals with those from fission spectra will be published in the DAFS Quarterly and subsequently at the Albuquerque meeting.

TITLE:

Effect of 14-MeV Neutron Irradiation on Loss Tangent of Aluminum Oxide

INVESTIGATORS:

J. D. Fowler, Jr. and F. W. Clinard, Jr.

AFFILIATION:

Los Alamos National Laboratory

SPONSORING PROGRAM:

Department of Energy, Office of Fusion Energy

SUMMARY:

Radio frequency (rf) windows must retain their transmissivity (as represented by a low loss tangent) after exposure to low-to-moderate neutron fluences to avoid excessive energy absorption, thermal stresses, and consequent structural failure. Earlier work has shown that loss tangent of single-crystal aluminum oxide (Al_2O_3), a prime candidate material for this application, is significantly degraded at some frequencies by irradiation at room temperature to 10^{21} to 10^{22} spallation n/m^2 (similar to a fast neutron spectrum). After a dose of 5×10^{21} n/m^2 (interpolated), loss tangent was increased by roughly an order of magnitude at 2×10^4 Hz but showed no increase from the unirradiated value of 3×10^{-5} at 2×10^6 Hz.

Two samples of the same material were irradiated to 5×10^{21} n/m^2 (14 MeV) at RTNS-II to determine the spectral dependence of damage. To date, one of these samples has been measured. Results show a slightly higher loss tangent at 2×10^4 Hz than that observed after spallation neutron irradiation, with a factor of two increase at 2×10^6 Hz.

Damage calculations by Robinson and optical absorption measurements by Evans and Stapelbroek indicate that roughly twice as much damage is expected from 14-MeV neutrons as from fission neutrons in Al_2O_3 . Anion vacancies are responsible for the increase in optical absorption under either irradiation condition. In the present work the observed spectral dependence of damage

implies that different types of resonance-absorption centers are formed depending on neutron energy. These results show that spectral effects must be taken into account in considering radiation damage in rf window materials. At the present time there are no plans for further irradiations at RTNS-II in support of this work.

PUBLICATION STATUS:

The results described here have been submitted to the Fifth Annual Progress Report on Special Purpose Materials for Magnetically Confined Fusion Reactors (in press). Further details can be obtained by contacting the investigators.

TITLE:

Effect of 14-MeV Neutron Irradiation on MACOR Glass-Ceramic

INVESTIGATORS:

J. D. Fowler, Jr. and F. W. Clinard, Jr.

AFFILIATION:

Los Alamos National Laboratory

SPONSORING PROGRAM:

Department of Energy, Office of Fusion Energy

SUMMARY:

MACOR* machinable glass-ceramic has been proposed as the electrical insulator for neutral-beam-injector sources where the lifetime neutron dose is expected to be 10^{23} n/m². The spectrum will be relatively hard at this location because of neutron streaming.

This material consists of mica platelets in a borosilicate matrix, a microstructure that imparts its property of machinability. Both phases are silicates and are therefore potentially sensitive to low irradiation levels. In earlier work, irradiation studies of MACOR were carried out at room temperature to 10^{22} n/m² (14 MeV). Results showed little change in density, strength, thermal conductivity, and post-irradiation electrical resistivity, but TEM examination revealed apparent early stages of microstructural damage. This suggests that physical property changes at an order of magnitude higher dose may be significant.

Additional disk-shaped samples have been submitted to RTNS-II for irradiation to higher fluences. These samples are stacked so that the rearmost will receive roughly a 10 times lower dose than will that at the front, allowing dose-dependence of property changes to be determined. Measurements will include electrical resistivity, swelling, and microstructural changes. During calendar year 1982, a fluence of 5×10^{22} n/m² was achieved, and continued irradiation to a dose of 10^{23} n/m² has been requested.

*Code 9658. MACOR is a product of Corning Glass Works, Corning, NY.

To complete this work, samples of a bend-bar configuration suitable for strength and fracture toughness measurements are needed. Therefore, approval for a second irradiation test to 10^{23} n/m² has been requested.

PUBLICATION STATUS:

The earlier work at 10^{22} n/m² has been published: J. D. Fowler et al., J. Nucl. Materials 103-104, 755 (1981). Further information on the current irradiation test can be obtained from the investigators listed above.

TITLE:

RTNS-II Irradiation (14-MeV Neutron) of Ceramic-Insulated Instrument Components in Support of FMIT Test-End Design

INVESTIGATOR:

C. P. Cannon

AFFILIATION:

Hanford Engineering Development Laboratory

SPONSORING PROGRAM:

Fusion Materials Irradiation Test (FMIT) Project

SUMMARY:

RTNS-II irradiations were conducted in August 1982 in support of specifying instrument components for the Fusion Materials Irradiation Test (FMIT) test-end region. State-of-the-art mineral-insulated instrument cables and five thermocouple types were irradiated to a neutron fluence of $1.76 \times 10^{18} \text{ n/cm}^2$. The cables were fabricated with a special processing that controlled contaminants in the cable insulation. (The cable backfill gas was high purity N_2 .) This was done to minimize "masking" of radiation effects by impurities. The cable insulation resistance at elevated temperature was measured to verify that contaminants had been sufficiently controlled: 10^9 ohm-m at 650°C was achieved.

No measurable changes were observed in post-irradiation testing on any of the components except W/Re thermocouples. Insulation resistance, capacitance, dissipation factor, and pulse breakdown voltage threshold were measured on MgO , Al_2O_3 , and SiO_2 cable types. The calibration of T, J, R, and K thermoelement types remained unchanged to within 1°C over the irradiated length. Also, the W/Re thermocouples showed a 7% decrease in calibration.

Any measurable changes in the electrical properties of a component irradiated to $1.76 \times 10^{18} \text{ n/cm}^2$ (14 MeV) in this test would have suggested that it was not suitable for fusion neutron service environments. On the basis of this irradiation, properly fabricated MgO , Al_2O_3 , and SiO_2 insulated cables

remain viable candidates for FMIT applications. The fact that no changes greater than 1°C were observed on thermocouple types T, J, R, and K suggests that good flexibility is available in the choice of thermocouple type as well. The decalibration of the W/Re thermocouple is of academic interest only since this type of thermocouple is not required for FMIT.

RTNS-11 irradiation to a fluence of $1.5 \times 10^{18} \text{ n/cm}^2$ is scheduled to evaluate ceramic-to-metal seal components. It has been established that fast-fission neutron fluences as low as 10^{17} n/cm^2 cause loss of hermeticity in seals that would otherwise remain hermetic in thermal neutron fluences greater than 10^{22} n/cm^2 . The planned irradiation will evaluate the relative effect of seal geometry, ceramic type, purity, and grain size, which are believed to be different for bulk ceramic materials compared to seal components.

The results to date are encouraging and give hope that there will be no drastic surprises in placing often trouble-prone instrument components into the unprecedented high intensity 14-MeV neutron environment of the FMIT test-end region.

PUBLICATION STATUS:

A paper on this subject will be published at the conclusion of irradiation effects study on ceramic-to-metal seal components.

TITLE:

14-MeV Radiation Studies

INVESTIGATOR:

W. L. Barmore

AFFILIATION:

Lawrence Livermore National Laboratory

SPONSORING PROGRAM:

Department of Energy, Office of Fusion Energy

SUMMARY:

Creep Testing at RTNS-II

The mechanical behavior of materials pertinent to the U.S. fusion energy program is being studied in-situ under 14-MeV neutron irradiation. In the in-situ creep experiment conducted during September 1982, effects of neutron irradiation on the creep of nickel were studied under both cyclic and steady flux conditions near $0.3 T_m$ and stresses to 280 MPa. Stress and flux effects on creep rate were examined. Tensile creep properties were measured at various conditions of stress, strain, temperature, and flux, using one or a combination of the following tests: standard creep, stress relaxation, cyclic flux, and cyclic temperature.

Abrupt changes in creep rate produced in nickel near $0.3 T_m$ and stresses of ~ 250 MPa during neutron-beam cycling are attributed to fluctuating defect concentrations and partitioning in the microstructure. Since creep rates are determined from overall load column expansion during testing, thermal expansion effects caused by neutron-beam heating must be subtracted from the total expansion to obtain the component due to tensile creep. For nickel specimens, thermal expansion contributions to the apparent creep rate are $\sim 60\%$ of the observed expansion effect.

The effect of a 14-MeV neutron flux level on the change in creep rate of nickel is apparently nonlinear for the experimental conditions in this study. The creep behavior of nickel near a homologous temperature of $0.3 T_m$ is controlled by an intermediate temperature mechanism where the apparent activation energy for creep is a function of the applied stress; it can be described by a general equation of the type:

$$\dot{\epsilon} = f(S) g(F) \exp \{[-\Delta H + (\sigma - \sigma_B)]/RT\} .$$

The 14-MeV neutron irradiation of $9 \times 10^{15} \text{ n/m}^2/\text{s}$ ($\sim 3.4 \times 10^{-9} \text{ dps/s}$) for 2000 s on nickel near $0.3 T_m$ produces significant changes in the microstructure of nickel and hardens the material to plastic deformation by $\sim 25\%$ compared to base line material. Data from recent in-situ experiments are being analyzed and interpreted using current creep and fracture theories. Constitutive relations are being developed that will describe creep for the selected materials under various conditions of stress, strain, temperature, and neutron flux.

PUBLICATION STATUS:

W. Barmore, A. Ruotola, E. Raymond, and A. Mukherjee, "Effects of 14-MeV Neutron Irradiation on Creep of Nickel and Niobium," in Symposium on Radiation Damage for Fusion Reactors at St. Louis, MO, October 25-28, 1982; to be published in J. Nucl. Mater. (1983).

U.S. SECONDARY EXPERIMENTS
**(Department of Energy/
Non-Office of Fusion Energy)**

TITLE:

Antireflectivity Using Etched Nuclear Tracks

INVESTIGATOR:

R. W. Hopper

AFFILIATION:

Lawrence Livermore National Laboratory

SPONSORING PROGRAM:

Laser Program

SUMMARY:

We have been developing gradient-index antireflective surfaces on fused silica (and other optical materials) using etched nuclear tracks. The main potential application is to the next generation of high power laser fusion systems, including the Nova system now under construction at LLNL. Nova will use harmonic conversion to convert 1.06- μm laser light to 0.53- μm (2 ω) and 0.35- μm (3 ω) light. Fused-silica focusing lenses will be required for adequate transmission of the ultraviolet 3 ω light, and it is highly desirable that these lenses be antireflective at 2 ω and 3 ω for three reasons. First, the transmission increase enhances the energy arriving at the target. Second, any reflections back down the amplifier chain are amplified and focused, resulting in damage to optical components. Finally, reflections within the lens can concentrate and cause damage. Broadband antireflectivity can be achieved with multilayer evaporative coatings, but these are susceptible to damage by the intense laser light. Standard etching techniques used to produce antireflective surfaces on multicomponent optical glasses are not applicable to fused silica. Our prime-candidate antireflectivity method for Nova is a sol-gel coating process being developed by Westinghouse, but the method is not yet proved. The etched nuclear track method provides an alternative to the sol-gel process.

When highly energetic ions of sufficient atomic number (nuclear particles) impinge on a dielectric, they form a track of damage roughly 5 nm in diameter and 20 μ m long. Because the atoms in the nuclear tracks are displaced to high energy positions, the tracks are chemically more reactive than the undamaged material, and the material in the track etches at a higher rate than the undamaged material.¹ In the case of glasses, etching produces conical pits having a cone angle related to the ratio of the two etching velocities. By subjecting a glass optical component to a high fluence of nuclear particles and subsequently etching it, a surface layer is produced having a porosity that varies through its thickness. Thus, the refractive index diminishes smoothly from the bulk glass to the air, and the surface can be broadband antireflective.

The tracks must be etched deeply enough compared with the wavelength of the light to give an adequate transition thickness ($\sim \lambda/2$) and yet have a small enough cross section so that diffuse scattering is small. These conflicting requirements imply that the cone angle of the etched track must be small. For most glasses, which are already disordered, the difference in etching velocity between the track and the undamaged material is not very large, so the cone angle tends to be large. For silica, the cone half-angle can be approximately 12 to 15° with suitable etchants.

We use fission fragments to create the tracks. A 25- μ m-thick sheet of 92% ²³⁵U foil is placed against the glass, packaged in a suitable container, and placed in a polyethylene box; then the assembly is situated in the target chamber of RTNS-11 as an add-on experiment. One end of the polyethylene box is ~15 cm thick and is directed toward the source; it serves to thermalize most of the neutrons in order to capitalize on the high capture cross section of ²³⁵U for thermal neutrons. After a sufficient exposure (typically two weeks), the sample is removed. Its radioactivity is dominated by the beta radiation from the fission fragments imbedded near the surface of the glass and decays as $t^{-1.1}$; it is reasonably innocuous after approximately one week.

¹R. L. Fleischer, P. B. Price, and R. M. Walker, Nuclear Tracks in Solids (University of California Press, Berkeley, 1975).

The glass is then etched in a weak acid containing hydrofluoric acid. Should the method be applied to the large (74-cm-diam) Nova focusing lenses, these would simply be placed along the walls of the target chamber and utilize the background neutron flux in which case the exposure time would probably be a few months.

Dozens of samples have been exposed to date. A degree of antireflectivity is easily obtained, but diffuse surface scatter is a severe problem at 3 μ . Although the specular reflectivity can be made low, it seems unlikely that transmittances in excess of 98% would be possible; however, this may be adequate for Nova. The experiments are continuing.

PUBLICATION STATUS:

The work will be published when the results are adequate. A brief internal status report is available from the investigator.

TITLE:

Phase-Matched Second-Harmonic Generation in Potassium Dihydrogen
Phosphate After Neutron Bombardment

INVESTIGATORS:

M. A. Summers and M. S. Singh

AFFILIATION:

Lawrence Livermore National Laboratory

SPONSORING PROGRAM:

Nova, ICF Program

SUMMARY:

The sensitivity of angle-tuned phase-matched second-harmonic generation (SHG) in potassium dihydrogen phosphate (KDP) to 14-MeV neutron irradiation was investigated. Initial measurements were made in late 1980 of the optical transmission and residual radioactivity. In June 1982, additional measurements were made to observe changes in the indices of refraction as they affect the SHG phase-matching orientation in this negative uniaxial crystal.

PUBLICATION STATUS:

The early measurements of transmission and radioactivity were reported in the Laser Program Annual Report 1980, pp. 2-166. The recent measurements of the effect on phase-matching orientation have not been reported.

TITLE:

Median Residence and Dispersion Times for Fluidization of Crushed Oil Shale

INVESTIGATOR:

R. G. Mallon

AFFILIATION:

Lawrence Livermore National Laboratory

SPONSORING PROGRAM:

Department of Energy

SUMMARY:

The motion of particles in a fluidized bed of crushed oil shale was tracked by means of radioactivation of a small sample of the shale. A group of scintillation detectors followed the motion of the activated material. The radionuclide used was ^{28}Al , which has a half-life of 2.3 min. Irradiation was by means of a pneumatic transfer system. A total of 240 irradiations were performed, each with a duration of about 10 min. It was found that this method is very useful in studying fluidized beds. This group of experiments is now complete.

PUBLICATION STATUS:

Results have been published as Lawrence Livermore National Laboratory Report UCRL-88493.

TITLE:

Preparation of ^{74}As

INVESTIGATOR:

H. G. Hicks

AFFILIATION:

Lawrence Livermore National Laboratory

SPONSORING PROGRAM:

Nuclear Weapons

SUMMARY:

One gram of As_2O_3 was irradiated ("add-on" with another sample) to produce ^{74}As tracer for counter calibration and separation chemistry investigation. This was a one-shot process from which there will be no publication.

TITLE:

Isotopic Fractionation in Meteorites

INVESTIGATOR:

R. J. Borg

AFFILIATION:

Lawrence Livermore National Laboratory

SPONSORING PROGRAM:

Department of Energy, Office of Basic Energy Sciences

SUMMARY:

We have had several irradiations of mineralic materials and inorganic salts. The thrust of this research is to answer questions about the mobility of rare gases (Ne, Ar, Kr, Xe) in minerals, a phenomenon of importance in both age dating and solar system cosmology.

Minerals thus far irradiated include:

Sodalite ~ $\text{Na}_4(\text{AlSiO}_4)_3\text{Cl}$ (2 varieties)

Albite ~ $\text{NaAlSi}_3\text{O}_8$

Enstatite ~ MgSiO_3

Forsterite ~ Mg_2SiO_4

The average fluence was $\sim 10^{17}$ neutrons as determined by Fe foil dosimetry. The relevant nuclear reactions are

The irradiated minerals were in the form of size $50 \pm 10 \mu$ diam powders encapsulated in quartz ampules. Diffusion coefficients will be calculated from the rate of outgassing as measured by mass spectrometry.

In order to determine the integrated production cross sections for Ne, Ar, and Xe, five samples--NaCl, CsCl, KCl, $\text{Ba}(\text{NO}_3)_2$, and Mg--were irradiated. Again Fe foil dosimetry was employed, and all samples were encapsulated in quartz ampules. Isotopic ratios will be determined by mass spectrometry. The relevant nuclear reactions are

<u>Ne Production</u>	<u>Ar Production</u>
$^{23}\text{Na} (n,2n) ^{22}\text{Na} \xrightarrow{\beta^+} ^{22}\text{Ne}$	$^{41}\text{K} (n,np) ^{40}\text{Ar}$
$^{23}\text{Na} (n,np) ^{22}\text{Ne}$	$^{40}\text{K} (n,p) ^{40}\text{Ar}$
$^{23}\text{Na} (n,\alpha) ^{20}\text{F} \xrightarrow{\beta^-} ^{20}\text{Ne}$	$^{40}\text{K} (n,np) ^{39}\text{Ar}$
$^{25}\text{Mg} (n,\alpha) ^{22}\text{Ne}$	$^{39}\text{K} (n,p) ^{39}\text{Ar}$
$^{24}\text{Mg} (n,\alpha) ^{21}\text{Ne}$	$^{39}\text{K} (n,np) ^{38}\text{Ar}$
<u>Xe Production</u>	
$^{133}\text{Cs} (n,np) ^{132}\text{Xe}$	
$^{133}\text{Cs} (n,p) ^{133}\text{Xe}$	
$^{137}\text{Ba} (n,\alpha) ^{134}\text{Xe}$	
$^{135}\text{Ba} (n,\alpha) ^{132}\text{Xe}$	
$^{134}\text{Ba} (n,\alpha) ^{131}\text{Xe}$	
$^{132}\text{Ba} (n,\alpha) ^{129}\text{Xe}$	

PUBLICATION STATUS:

Results of this work will be submitted to a refereed journal.

TITLE:

Effects of 14-MeV Neutron Bombardment on the Electrical Properties of
Small-Geometry Semiconductor Devices and Integrated Circuits

INVESTIGATORS:

R. A. Hartmann and J. R. Srouf

AFFILIATION:

Northrop Research and Technology Center

SPONSORING PROGRAM:

Defense Nuclear Agency

SUMMARY:

In this ongoing program, the effects of 14-MeV neutrons on the electrical properties of small-geometry semiconductor devices and integrated circuits are being assessed. Specifically, the possibility that a disordered region produced by a single neutron interaction in a critical region of a device might catastrophically degrade the properties of that device is being investigated. This issue is relevant at present because integrated circuit geometries are being reduced significantly.

PUBLICATION STATUS:

Our early work in this area was published in the IEEE Transactions on Nuclear Science, Vol. 28, December 1980, pp. 3968-3974. Work performed during 1982 will appear in the final report for Contract DNA001-82-C-0055 (in preparation).

TITLE:

The Nature of 14-MeV Neutron Damage in Silicon

INVESTIGATOR:

G. Woolhouse

AFFILIATION:

ARACOR, Sunnyvale, CA

SPONSORING PROGRAM:

Defense Nuclear Agency (DNA)

SUMMARY:

We have carried out 14-MeV neutron irradiation of silicon samples to determine the nature of the damage in silicon and particularly to distinguish between two models that have been put forward: the small cluster model and the large void model. Our transmission electron microscopy analyses indicate that the small cluster model is appropriate in this case. Some work is ongoing in this area.

PUBLICATION STATUS:

This work was included in a report to DNA. No written report has been made. Future plans are currently uncertain.

FOREIGN

TITLE:

14-MeV Neutron Damage in Vanadium and Nickel

INVESTIGATOR:

D. Kaletta

AFFILIATION:

Kernforschungszentrum Karlsruhe

SPONSORING PROGRAM:

Materials Behavior Under Irradiation

SUMMARY:

The objective of this work was to measure defect yields and determine the character of loops produced in pure nickel and vanadium. Transmission electron microscope specimens of both materials and tensile specimens of vanadium were irradiated at room temperature to $8 \times 10^{17} \text{ n/cm}^2$.

The loop analysis is in progress. It is clear that loop production is very different in the two metals. Nickel exhibits very few small loops, but vanadium has many loops of large size. There is evidence for sub-cascade and lobe formation.

Tensile measurements have not yet been made. They will cover a test temperature range of 600 to 1000°C for comparison with earlier alpha-irradiated specimens.

PUBLICATION STATUS:

A paper describing this work will be submitted for presentation at the Third Topical Meeting on Fusion Reactor Materials, Albuquerque, NM, September 1983.

TITLE:

Production of ^{93m}Nb Activity for Instrument Calibration

INVESTIGATORS:

G. Winkler and H. Vonach

AFFILIATION:

Institut für Radiumforschung und Kernphysik der Österreichischen Akademie der Wissenschaften und der Universität Wien

SUMMARY:

A one-shot irradiation was performed with $\sim 10^{17}$ 14-MeV n/cm² in April 1982 to produce a pure ^{93m}Nb activity in two thin niobium foils (diameter 20 mm, thickness 0.0125 mm, purity 99.9%, ~ 14.5 mg) via the reaction $^{93}\text{Nb} (n, n'\gamma) ^{93m}\text{Nb}$. The activated Nb foils are intended for the calibration of a solid-state x-ray detector to provide an energy differential measurement of the $^{93}\text{Nb} (n, n'\gamma) ^{93m}\text{Nb}$ cross section (primarily around 3 MeV), which is important for reactor dosimetry applications.

To reliably calibrate an x-ray detector to measure the Nb x-radiation from internal conversion of ^{93m}Nb induced in thicker samples of niobium, a pure activity of ^{93m}Nb was required incorporated in a low-mass Nb-foil. This foil can be used to measure self-absorption corrections by means of simulating the activity sitting in different layers of a larger sample. Activation in a reactor spectrum does not provide an interference-free ^{93m}Nb with commercially available Nb foils. In particular, Ta impurities more than a few ppm cause a problem, resulting in ^{182}Ta due to neutron capture. The low-energy γ -radiation and x-radiation of ^{182}Ta ($T_{1/2} = 115$ d) induces characteristic Nb x-radiation in thicker samples.

The x-ray and γ -ray spectrum of the irradiated foils was checked by means of both a Si(Li) and a Ge(Li) detector. After a waiting time of more than six months, the ^{92m}Nb activity ($T_{1/2} = 10.15$ d, Zr X-radiation) from the $^{93}\text{Nb} (n, 2n) ^{92m}\text{Nb}$ reaction had decayed to a negligible amount, and the x-ray peaks at 16.58 keV (K_{α}) and 18.66 keV (K_{β}) from the decay of ^{93m}Nb ($T_{1/2} = 13.6$ a, internal conversion) remained undisturbed. Two

additional x-ray peaks from tungsten X_L -radiation could be identified, which stem from ^{182}Ta were the only contaminants that could be detected resulting from nuclear reactions on impurities (approximately 5-ppm Ni and approximately 500-ppm Ta) in the sample. The level of contamination is low enough not to produce any interference for the efficiency calibration procedure.

PUBLICATION STATUS:

At the present time there is no publication available.