RTNS-II 1983 Annual Report
Joint U.S.-Japan Sponsorship

Irradiations at the
Rotating Target
Neutron Source-II

ON THE COVER:
When irradiated at RTNS-II with 14-MeV neutrons, samples of pure gold show cascade defects under TEM. The sample at the top is shown before irradiation at a magnification of 50,000. The center sample was irradiated below 20 K (8 x 10^16 n/cm^2), then transferred to TEM without warmup and observed at 160 K. Magnification in this photo is 100,000. The bottom shows results at 300 K (1.5 x 10^16 n/cm^2) at 100,000 magnification. Photos are courtesy of Professor Y. Shimomura, Hiroshima University.

Lawrence Livermore National Laboratory
FOREWORD*

This is the second annual report summarizing irradiation experiments and operations at RTNS-II. It covers calendar year 1983 and includes reports on all irradiations, non-fusion as well as fusion, and on utilization of Monbuso's transmission electron microscope (TEM) at RTNS-II.

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

Clint Logan
RTNS-II Facility Manager

*Work performed under the auspices of the U.S. Department of Energy 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 Japan Monbusho agreed to jointly support the operation and development of RTNS-II, and to share in its utilization.

RTNS-II was created to provide a D-T 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:

- Acquire direct engineering data for near-term confinement experiments and for materials that will see moderate neutron dose in future reactor systems.
- Measure production rates for transmutants and radioactivity, and develop appropriate radiation-resistant instrumentation for fusion systems.
- Contribute to understanding neutron spectral effects on radiation-induced property changes.

RTNS-II contains 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 ~340 keV by a Cockroft-Walton power supply. We presently deliver 130 mA of ~370 keV deuterons to the target in a beam spot size ~1 cm in diam.

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. In order to limit the peak temperature reached from 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 utilizing a broad array of research tools. In this work, RTNS-II has a unique role. It is the world's only 14-MeV neutron source dedicated to materials research.
Copper alloys of several compositions are being considered for use in manufacturing RTNS-II targets. These alloys contain elements that are difficult to assay accurately with conventional techniques. Some of the alloying elements are present in solid solution and as insoluble oxides, eliminating conventional wet chemical analysis as a viable method. We exposed sample disks of six different materials along with calibration foils of Al, Fe, Mg, Zr, Ag, and Cr. Dosimeters were Nb. All samples and foils were 1.6 cm in diam. Mylar separators prevented contamination from direct recoil "hot atom transport."

Fast neutron activation is a useful method for determining total content of the elements listed. It is not appropriate, however, to assay for all elements in a single irradiation. Aluminum, for example, produces a relatively short-lived radioactive isotope, and therefore should be measured quickly after a short irradiation. Others require longer irradiation times to build sufficient activity. This technique is not easy or quick, so it is not a substitute for more conventional techniques where they are possible.

We had a second purpose for these measurements. The quantity of a radioactive isotope present in the mylar separators is a direct measurement of the mean projected range of the hot atom recoiling from a neutron interaction. For these measurements, the sample/source geometry resulted in each sample intercepting neutrons over a large solid angle, so the implied recoil range is averaged over a large angular range for the recoiling.
radioactive ion. Therefore, we have not reported these values. The technique could be applied in more appropriate geometry to measure hot atom transport (forward and backward) with respect to the angle of the incident neutron.

PUBLICATION STATUS:

No publication is planned.
Effect of 14-MeV Neutron Irradiation on Thread-Locking Compound

INVESTIGATOR:
C. M. Logan

AFFILIATION:
RTNS-II Facility, Lawrence Livermore National Laboratory

SUMMARY:
Proper functioning of the RTNS-II rotating target assembly depend on Locktite #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. Last year we prepared an add-on experiment to measure the response to 14-MeV neutrons. The results lacked plausibility.

We have now completed an improved version of this experiment. Bolts and nuts 0.25 inch in diam and 28 threads/inch were selected from a single lot number, cleaned, and assembled with Locktite #242. One week of curing time at room temperature was allowed before beginning irradiation. Five sets of ten fasteners were irradiated so that each of the ten within a set received the same neutron fluence. The five sets ranged in fluence from $3.9 \times 10^{15}$ n/cm$^2$ to $8.4 \times 10^{16}$ n/cm$^2$.

Values for post-irradiation breakaway torque are plotted in Fig. 1 for unirradiated controls and for the five irradiated sets. In most sets there was one value significantly higher than the other nine. The explanation for this is not obvious. Significant scatter is present in the data, even the unirradiated controls. Averages of each set are plotted as x's. No trend with fluence is revealed by this data. Our measurements for torque that were required to maintain fastener rotation after breakaway also contained large scatter and no clear dependence on fluence.
Fig. 1. Breakaway torque vs neutron fluence for Locktite #242.
Based on this data, we conclude that Locktite #242 can be used on RTNS-II rotating assemblies without concern for neutron-induced degradation.

PUBLICATION STATUS:

No publication is planned.
Neutron Monitor Calibration

D. W. Heikkinen

RTNS-II Facility, Lawrence Livermore National Laboratory

Neutron production for both neutron sources at RTNS-II is monitored using proton recoil counters and ionization chambers. These are used for instantaneous and cumulative production results and also to provide the time history of an irradiation. These neutron production monitors are periodically calibrated using Nb dosimetry foils. In general, the cross calibrations agree to within a few percent.

We are planning to miniaturize the proton recoil counters, making them less sensitive to background.

The neutron monitor calibration records are maintained at RTNS-II.
TITLE:
Irradiation Effects on Carbon Fiber/Epoxy Composite Material

INVESTIGATOR:
T. M. Vercelli

AFFILIATION:
RTNS-II Facility, Lawrence Livermore National Laboratory

SUMMARY:
Proper functioning of the 50-cm rotating target assembly at RTNS-II depends on a carbon fiber/epoxy ring mated to the target cone for reinforcement during rotation. Response of this material to radiation is unknown. We prepared an add-on experiment to measure the degradation of tensile strength of the carbon fiber/epoxy material. The expected lifetime dose to this carbon fiber/epoxy composite in operation on RTNS-II is $2 \times 10^{17}$ n/cm$^2$ for three years of operation.

The experiment resulted in a measured fluence on three test specimens of $3.0 \times 10^{17}$ n/cm$^2$, $4.7 \times 10^{17}$ n/cm$^2$, and $2.9 \times 10^{17}$ n/cm$^2$. The test specimens are composite hoops approximately 6 inches in diam and 0.25 inches wide, with 0.062-inch wall thicknesses. The irradiation was passive. Dosimetry foils were required.

There were no in-situ measurements. Post-irradiation measurement of hoop burst strengths will be performed next year on the three irradiated rings, along with unirradiated control specimens.

PUBLICATION STATUS:
A Laboratory report is planned.
A. Introduction

Many experimental studies of neutron irradiation to superconductors have been done using fission reactors. Since Monte Carlo calculations predict that the neutron energy spectrum in the superconducting magnet region in a fusion reactor is similar to that in a fission reactor, one might think it sufficient to take the neutron irradiation data for the fluence up to $10^{18} \sim 10^{19}$ n/cm$^2$ with the fission neutron spectrum. From the practical point of view, however, it is insufficient as a simulation or irradiation under fusion reactor conditions, because:

1. The streaming neutrons in the SCM may result in a higher flux than $10^9$ n/cm$^2$ s by several orders of magnitude.
2. The spectrum of streaming neutrons should be harder, i.e., the fraction of 14-MeV neutrons must be higher than that of unstreamed neutrons. Another important factor for the irradiation data is the irradiation temperature, if the superconducting properties are sensitive to the lattice defects, which may anneal out at elevated temperatures.

The main objective of this using 14-MeV neutrons from RTNS-II is to in situ irradiation data that involves (1) the spectrum effect, i.e., fission-fusion correlation (2) cryogenic irradiation. Since fusion neutron and cryogenic irradiation is very expensive, our intent has been to establish the appropriate methodology for the simulation of the fusion condition.
E. Irradiation Equipment

The preparation procedure of a pig-nose cryostat was as follows:
1. Heilitran (LT-3B-110) was purchased by the Institute of Plasma Physics Laboratory from Air Products Co., U.S.A. (Dec. 1982).
2. This was operated three times at Osaka University, Japan and transferred to Lawrence Livermore National Laboratory (LLNL) (January through March 1983).
3. Thermal shield and cold tip were modified (June 1983).
4. Three current lead (30 A) and 18 thin potential leads were fixed to the cryostat (July 1983).
5. Two carbon glass resistors (Lake Shore) and two copper-constantan thermocouples were fixed to the cold finger.
6. Seven samples (four from Japan and three from the U.S.) were set on the cold stage with iron wires, which are necessary for the neutron dosimetry.

C. Experimental Results

On September 26, 1983, the fluence reached $1 \times 10^{18}$ n/cm$^2$, and successive changes of the critical current density were successfully measured at 8 and 12 T. Figure 2 shows the $J_c/J_{co}$ against 14-MeV neutron fluence. The contrast between BNL monofilament Nb$_3$Sn and in situ Nb$_3$Sn is great and is now under analysis.

D. Conclusion

It is very important to notice that the present pig-nose irradiation system developed by Mike Guinan of LLNL over the past several years has shown that the highest performance in the world for the cryogenic irradiation with 14-MeV neutrons is the high fluence of $1 \times 10^{18}$ n/cm$^2$.

(1) Methodology
A continuous flow type cryostat (modified Heilitran LT-3B-110E) is useful as a strong tool for the simulation of fusion magnet conditions with cryogenic $1^\text{e}$-TeV irradiation. The obtained fluence
Fig. 2. Change in $J_c$ against neutron fluence.
of $1.3 \times 10^{18}$ n/cm$^2$ is close to what a fusion magnet would have for the reactor life of 20 years. The obtained material data is useful as the basis of magnet design.

(2) Superconductors

Different fabrication methods with Nb$_3$Sn show different behaviors. In situ Nb$_3$Sn showed a 5% decrease in $J_c$ at 12 T at $1.0 \times 10^{18}$ n/cm$^2$. Yet monofilament Nb$_3$Sn showed no degradation at that level. Detailed behaviors associated with pinning plot and Kramer plot will be discussed elsewhere. Amorphous Mo-Si shows little change even after 14-MeV irradiation of $1.0 \times 10^{18}$ n/cm$^2$.

**PUBLICATION STATUS:**


SUMMARY:

A. Introduction

The titanium tritide targets that are now being used are prepared by the vacuum deposition of titanium on a copper substrate. This coating is then tritiated. However, these targets are limited in their ability for long-term operation and long-term storage. This is probably because of low chemical stability of the titanium tritide at relatively high temperatures and weak adhesion of the titanium coating to the substrate surface.

Therefore, we hope to improve the neutron yield performance of the titanium tritide target and to develop a metal tritide target using a metal such as yttrium, erbium, or scandium rather than titanium.

It is also well known\textsuperscript{1,2} that the ion sputtering technique can generally serve as a method to make solid metal coatings with tight bonding to many substrates, mainly because of the bombardment of the substrate surface by the sputtered atoms, the energies of which are many times higher than could be exhibited by thermally evaporated atoms.

Thus, we can expect that sputtered titanium coatings will be obtained that bond more tightly to copper substrates than those prepared by vacuum deposition. In this work some experiments were carried out to examine the applicability of an ion sputtering technique as a method for making metal tritide coatings that are more tolerable to the deuteron beam irradiation than the normal vacuum evaporated titanium coatings.
B. Conclusion

When titanium is coated by the radio-frequency sputtering technique on a carefully cleaned copper surface, the titanium film obtained shows tighter bonding than that of thermal evaporation.

Hydriation of the titanium film causes a considerable reduction of the film adhesion to the substrate. Formation of a composite layer of titanium and copper at the interface may solve this problem. In that case it is necessary to study the effect of the composite layer's thickness on the target performance.

REFERENCES:

1. R. W. Berry et al., Thin Film Technology (Krieger, 1979).

PUBLICATION STATUS:

There is a paper pending for Osaka University.
TITLE:
A Measure of Neutron Energy Spectra from Lithium Chloride Sample by Multiple-Foil Activation Technique

INVESTIGATOR:
C. Ichihara

AFFILIATION:
Research Reactor Institute, Kyoto University

SUMMARY:
An integral experiment of isotopically separated lithium chloride was performed using the multiple-foil technique and the 14-MeV neutron source at RTNS-II.

Lithium chloride powder (95.5 at.% Li, 97.5 at.% Li) was enclosed in cylindrical aluminum containers 308 mm long and 292.5 mm in diam (Fig. 3). The activation foils were Ni, Cu, Au, Ta, Fe, Co, and Ti, and they were set at the middle point and the backplate of each container. The containers were located 300 mm apart from the rotating target of RTNS-II, at a 90-deg angle from the deuteron beam axis (Fig. 4). They were irradiated for 85 hours.

Gamma-ray measurements were performed using pure germanium detectors and multi-channel analysers. The reaction rates for the activation foils were deduced from these results. The unfoldings of the energy spectra are now in progress using STAY'SL and NEUPAC unfolding codes.

The neutron energy spectrum for each Li sample was calculated with a Monte Carlo transport code (TART) using the CRAY-I computer. These results will be compared with the obtained spectra.

PUBLICATION STATUS:
This work is reported in the Annual Research Report of the Japanese Contribution for U. S.-Japan Collaboration on RTNS-II Utilization (1983).
Fig. 3. Aluminum container with dimensions given in mm.
Fig. 4. (a) Geometrical configuration of the irradiation (from above), and (b) picture of the irradiation.
TITLE:

A Study for Using Magnesium Oxide Crystals as Fast Neutron Dosimeters

INVESTIGATOR:

C. Ichihara

AFFILIATION:

Research Reactor Institute, Kyoto University

SUMMARY:

Color centers are produced by the neutron irradiation on single crystals of magnesium oxide. The results of the neutron irradiation on magnesium oxide crystals at the Kyoto University reactor showed that the optical absorption of the irradiated crystals increases with an increasing neutron dose. Such a characteristic can be used to measure the neutron doses. These doses are free from correction for the fluctuation of the neutron intensity, the decay of the activities, and other problems characteristic to the foil activation method.

Thin pieces of nominally pure single crystals of magnesium oxide were irradiated with 14-MeV neutrons at RTNS-II. The neutron fluences were measured with Fe foils and were $1.9 \times 10^{15}$, $6 \times 10^{16}$, $8 \times 10^{16}$, and $3.4 \times 10^{17}$. The optical absorption was measured with a spectrophotometer at the liquid nitrogen temperature at Kyoto University. The obtained absorption spectrum is given in Fig. 5. The relations between the optical absorption coefficient and the 14-MeV neutron doses for three wavelengths are illustrated in Fig. 6, together with one of fission neutrons. It is encouraging that the absorption for each wavelength increases linearly on this figure. However, further irradiations are necessary to see the maximum dose where the optical absorption saturates.

PUBLICATION STATUS:

This work is reported in the Annual Research Report of the Japanese Contribution for U. S.-Japan Collaboration on RTNS-II Utilization (1983).
MgO 14 MeV, RTNS-II
fluence: $8 \times 10^{16} \text{n/cm}^2$

Fig. 5. Typical optical absorption spectrum.
Fig. 6. Obtained absorption coefficients vs neutron fluences.
The Feasibility of a TLD as a Tritium Detector

INVESTIGATORS:
K. Saneyoshi and M. Ogawa*
D. Heikkinen and C. Logan**

AFFILIATION:
*Tokyo Institute of Technology
**Lawrence Livermore National Laboratory

SUMMARY:

A. Introduction

With the advance of research on nuclear fusion reactors, the study of tritium breeding in a blanket assembly has become very important. Measuring tritium is an essential technique in the study. There are two well-known methods, one using liquid scintillation counters and the other using gas-filled counters. Maekawa¹ has proposed a new method using thermoluminescent dosimeters (TLD). This method is based on self-irradiation caused by the β-decay of tritium that is produced in a TLD irradiated with neutrons. It is simpler than those described above because there is no chemical procedure and because of the short counting time. Recently there have been two experiments using self-irradiation: Maekawa² and Sharabati et al.³ Because of low neutron fluence (10¹² n/cm²) these two experiments had large errors caused by low counting rates.

The intense neutron source RTNS-II is suitable to perform this kind of experiment because higher fluences can be obtained in a short time period. This report describes an ongoing experiment; thus, a conclusion has not yet been reached, but neutron induced activities and some results of self-irradiation without any blanket assembly have been obtained.

B. Calculation of Tritium Production in a TLD Chip

The TLDs employed in this experiment are TLD600 and TLD700 chips, manufactured by Harshaw Chemical Co., Ltd. The properties of the TLD chips are:
Size: 3 x 3 x 0.9 mm

Number of Li/cm$^3$: $N_1 = 6.12 \times 10^{22}$ n/cm$^3$

Composition TLD600: $^6$LiF 95.6%, $^7$LiF 4.4%, (Mg, Ti 100-200 ppm)

Composition TLD700: $^7$LiF 99.99%, (Mg, Ti 100-200 ppm)

The tritium production $N_A$ in a TLD chip at a neutron energy of 14 MeV (i.e., without blanket) is calculated as follows:

$$N_A = nf[1 - \exp(-Na_A t)]$$ \hspace{1cm} (1)

where subscript $A$ is 6 for $^6$LiF and 7 for $^7$LiF, respectively, $f$ is the fluence (n/cm$^2$), and $\sigma_A$ is the cross section for tritium production at a neutron energy of 14 MeV. Then

$$\sigma_6 = 2.57 \times 10^{-26} \text{ cm}^2 \text{ for } ^6\text{Li}(n,\alpha)t$$

$$\sigma_7 = 3.07 \times 10^{-25} \text{ cm}^2 \text{ for } ^7\text{Li}(n,n'\alpha)t$$ \hspace{1cm} (2)

Substituting these values for Eq. (1), we obtain for $f = 10^{15}$ n/cm$^2$

$$N_6 = 1.27 \times 10^{10} \text{ n/chip}$$

$$N_7 = 1.52 \times 10^{11} \text{ n/chip}$$ \hspace{1cm} (3)

Using the composition described above and the decay constant of tritium ($1.79 \times 10^{-7}$ s$^{-1}$), the results are

9.1 x 10$^{-4}$ µCi/chip for TLD600

7.4 x 10$^{-3}$ µCi/chip for TLD700

Assuming a mean $\beta$ energy of 5.6 keV and that all of the energy is deposited in the chip, the doses caused by self-irradiation after a neutron irradiation of $10^{15}$ n/cm$^2$ and one month of waiting using 89.0 erg/g*R for TLD600 and 85.7 erg/g*R for TLD700$^1$ the following is expected:

36.7 erg/g*month (427 mR/month) for TLD600

299 erg/g*month (3.41 mR/month) for TLD700

These calculated doses obtained are sufficient to observe TLs produced by self-irradiation.
C. Experiment

Neutron Irradiation. Twenty-two TLD chips were irradiated with 14-MeV neutrons. The samples were divided into two groups. The first group (samples 1 through 5) was composed of only TLD 700 and irradiated for about 100 hours. The position of each sample of the first group was determined by the calculation to be within a neutron fluence range from $10^{13}$ to $10^{17}$ n/cm$^2$ and differed by a factor of ten from the second group. The measured fluences using the activations of 3-mm Nb foils, which are shown in Table 1 agreed with the calculated values very well, with the exception of sample 1. This sample was close to the neutron source, and the exact placement was difficult to determine. The samples of the second group (6 through 11) were placed so that the fluence of each sample was between those of the first group. The irradiation time of the second group was about 64 hours. The measured fluences are also shown in Table 1.

Table 1. Neutron fluence measured by Nb foils.

<table>
<thead>
<tr>
<th>Sample number</th>
<th>Type of TLD sample</th>
<th>Neutron fluence (n/cm$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>700</td>
<td>$1.47 \times 10^{17}$</td>
</tr>
<tr>
<td>2</td>
<td>700</td>
<td>$1.06 \times 10^{16}$</td>
</tr>
<tr>
<td>3</td>
<td>700</td>
<td>$1.03 \times 10^{15}$</td>
</tr>
<tr>
<td>4</td>
<td>700</td>
<td>$1.03 \times 10^{14}$</td>
</tr>
<tr>
<td>5</td>
<td>700</td>
<td>$0.98 \times 10^{13}$</td>
</tr>
<tr>
<td>6</td>
<td>600</td>
<td>$4.69 \times 10^{16}$</td>
</tr>
<tr>
<td>7</td>
<td>700</td>
<td>$4.44 \times 10^{16}$</td>
</tr>
<tr>
<td>8</td>
<td>600</td>
<td>$6.95 \times 10^{15}$</td>
</tr>
<tr>
<td>9</td>
<td>700</td>
<td>$6.99 \times 10^{15}$</td>
</tr>
<tr>
<td>10</td>
<td>600</td>
<td>$5.11 \times 10^{14}$</td>
</tr>
<tr>
<td>11</td>
<td>700</td>
<td>$5.11 \times 10^{14}$</td>
</tr>
</tbody>
</table>
The color of the samples, originally white, changed to yellow, orange, brown, and dark brown, with yellow being the lowest fluence and dark brown the highest. The irradiation damage in the TLDs causes a reduction in efficiency that may interfere with the self-irradiation method, but this damage can be recovered by annealing. Later, this problem will be described.

**Induced activities.** After irradiation, the gamma-ray spectra of samples 1 through 3 and 6 through 9 were measured. The spectrometer used was the Gamma Gage-GEM Ge(Li) spectrometer manufactured by EG&G ORTEC, Inc. The measurements were performed 2 days, 6 days, and 12 days after irradiation. Most of the gamma-rays observed were identified. The result measured 6 days after is shown in Table 2. The values of activities obtained contain scatter caused by statistical errors. The annihilation radiation of sample 1 is quite high, which is not now understood. The origin of $^7$Be is not understood, and the yield in TLD600 seems to be twice as high as in the TLD700.

Comparing the results to the calculated tritium yield, the number of Curies of impurities except in $^7$Be are less than 1% of the tritium. In the case of the $^7$Be, the decay is an electron capture, and 90% of them decay to a ground state. Thus, the energy deposit in the TLD is fairly small. Therefore, the influence of the induced activities can be neglected. In practice, a simple comparison of the number of Curies is not sufficient. We must consider the energy deposit for a decay. A more detailed analysis is now being performed.

**Self-irradiation.** Before storing in a low background environment, the TLD chips must be annealed to erase the TLs produced by neutron irradiation. On the other hand, annealing at high temperatures for long periods of time causes the release of tritium from the LiF. Even though Tabata et al. used 600 °C, our TLDs were first annealed at 300 °C for 1 hour and then at 100 °C for the next 20 hours, then read by a TLD reader, manufactured by Matsusita Electric Co., Ltd. The counts obtained did not decrease to the background level even after annealing them twice. Finally, the procedure used was to anneal at 400 °C for 1 hour and 120 °C for the next 20 hours, store in a low background environment for 2-1/2 days, and then read. This procedure was repeated after 11 days storage. The TLDs were annealed a third time.
Table 2. Induced activities 6 days after irradiation. Units are μCi/chip.

<table>
<thead>
<tr>
<th>Nuclide</th>
<th>7Be</th>
<th>24Na</th>
<th>46Sc</th>
<th>47Sc</th>
<th>48Sc</th>
<th>E+</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reaction</td>
<td>24Mg(n,p)</td>
<td>46Ti(n,p)</td>
<td>47Ti(n,p)</td>
<td>48Ti(n,p)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Half-life</td>
<td>53.3 days</td>
<td>15.0 hours</td>
<td>83.8 days</td>
<td>3.42 days</td>
<td>43.7 hours</td>
<td>3 days&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>Sample 1</td>
<td>1.6 x 10&lt;sup&gt;-2&lt;/sup&gt;</td>
<td>5.6 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
<td>2.1 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
<td>2.8 x 10&lt;sup&gt;-4&lt;/sup&gt;</td>
<td>9.1 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
<td>1.0 x 10&lt;sup&gt;-3&lt;/sup&gt;</td>
</tr>
<tr>
<td>2</td>
<td>1.8 x 10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>--&lt;sup&gt;b&lt;/sup&gt;</td>
<td>--</td>
<td>3.7 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
<td>--</td>
<td>6.1 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
</tr>
<tr>
<td>3</td>
<td>2.5 x 10&lt;sup&gt;-4&lt;/sup&gt;</td>
<td>--</td>
<td>--</td>
<td>1.5 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
<td>--</td>
<td>5.3 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
</tr>
<tr>
<td>6</td>
<td>2.6 x 10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>6.8 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
<td>--</td>
<td>3.1 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
<td>2.8 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
<td>1.2 x 10&lt;sup&gt;-4&lt;/sup&gt;</td>
</tr>
<tr>
<td>7</td>
<td>4.7 x 10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>7.8 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
<td>--</td>
<td>3.4 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
<td>3.8 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
<td>1.2 x 10&lt;sup&gt;-4&lt;/sup&gt;</td>
</tr>
<tr>
<td>8</td>
<td>3.9 x 10&lt;sup&gt;-4&lt;/sup&gt;</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>7.8 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
</tr>
<tr>
<td>9</td>
<td>8.9 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
<td>--</td>
<td>--</td>
<td>1.1 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
<td>--</td>
<td>1.9 x 10&lt;sup&gt;-5&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>a</sup> Obtained from measurement.
<sup>b</sup> Not observed.
read again after 1 day storage. The results obtained are shown in Fig. 7. In that figure, it appears that self-irradiation has occurred because of the increase in counts with fluence and storage time. This increase might also be caused by some effects of irradiation damage. To confirm self-irradiation, TLD700 chips irradiated with gamma rays to $10^6$ rad and the TLD600 chips irradiated with 14-MeV neutrons are to be measured by the same procedure used above.

The counts in Fig. 7 are not proportional to the fluence. This may be caused by the reduction in efficiency at a higher fluence. It might be necessary to anneal at a higher temperature and for longer periods. To follow this procedure we must know precisely the temperature and time dependence of tritium release from a TLD chip. A system similar to Tabata's is now being constructed to measure tritium release. This system is shown schematically in Fig. 8. It is capable of heating a TLD chip to 800 °C and capturing THO and TF in a trap cooled with liquid nitrogen. Then the amount of tritium is measured using a liquid scintillation counter.

REFERENCES:

2. H. Maekawa, JAERI-M 82-114 (1982).

PUBLICATION STATUS:

This work is reported in the Annual Research Report of the Japanese Contribution for U. S.-Japan Collaboration on RTNS-II Utilization (1983).
Fig. 7. TLD output after storage.
Fig. 8. Tritium detection system.
The second run for mainly pure metals and alloys was performed between January and March of 1963 by the Japanese material research groups together with Dr. E. R. Bradley of Pacific National Laboratory. The author took care of the irradiation and TEM observation at LLNL as a Japanese principal experimenter of the run.

The objective of this work is to evaluate the effects of cascade damage on microstructure evolution. In order to understand the damage accumulation under the fusion reactor circumstance, it is necessary to make clear the elementary processes of microstructure evolution by 14-MeV neutron irradiation. Highly heterogeneous defect production caused by high energy primary knock-on atoms is expected to be one of the major factors that determine the microstructure evolution.

Post-irradiation observation by TEM on some metals will be reported here. TEM specimens of a large variety of metals and alloys, such as pure metals, pure alloys, and practical alloys were prepared to reduce the general features of cascade damage and peculiarity of each material depending on its lattice structure, defect properties, and other characteristics.

The 14-MeV neutron irradiations were conducted at 200 and 400 °C by using a dual temperature vacuum furnace made by Hanford Engineering Development Laboratory. The maximum neutron fluence at the specimen position closest to the neutron source was $20 \times 10^{18}$ n/cm$^2$, or about 0.08 dpa.

After the neutron irradiation, about 40 specimens were electro-polished and successively observed by a TEM at LLNL.

One prominent feature of 14-MeV neutron irradiation damage in metals is an enhanced formation of interstitial loops along dislocation lines. This is
in contrast with the homogeneous damage caused by electron irradiation where nucleation of interstitial loops is suppressed along dislocation lines by the sink effects of the loops. Results suggest that most of the interstitial loops are formed heterogeneously, probably during the cascade damage events and their subsequent internal rearrangements. Once a nucleus is formed, it grows by absorbing "free" interstitials. If this occurs in the vicinity of a dislocation line, the loop grows quickly by snatching the interstitials that are flowing to the dislocation by a long range elastic interaction among them.

Such effects of cascades promote microstructure evolution; therefore, they might shift the void swelling peak to the higher temperature side.

Typical vacancy clusters formed in metals of low stacking fault energy are stacking fault tetrahedra (SFT). Their observed features suggest strongly that they were formed during cascade damage events and subsequent internal rearrangements.

It was clear from this work that SFT played a very important role for the initial process of microstructure evolution. Because their bias effect for point defects is weak, they have long lifetimes under the irradiation. Thus, excess interstitials, corresponding to the vacancies clustering as SFT, form their clusters (interstitial loops) and make their microstructure complicate.

Small voids were observed in Cu and Ni at 400 °C. They should play very important roles for the void swelling.

PUBLICATION STATUS:

Previous reports dealing with the subject follow.

Publications


Oral Presentations


RTNS-II

Japanese-Initiated Experiments
MACOR machinable glass-ceramic (a product of Corning Glass Works, Corning, NY) has been proposed as the electrical insulator for neutral beam injector sources, where the lifetime neutron dose is expected to be $10^{23}$ n/m$^2$. The spectrum will be relatively hard at this location because of neutron streaming.

Earlier irradiation studies at RTNS-II showed no major changes in structural and electrical properties of this potentially damage-sensitive silicate material after a dose of $10^{22}$ n/m$^2$ at room temperature. However, TEM examination revealed apparent early stages of microstructural damage. Additional samples in the form of stacked disks were submitted for graded irradiation to between $10^{22}$ and $10^{23}$ n/m$^2$. The target dose has now been achieved. Upon their return to Los Alamos National Laboratory (LANL) the samples will be evaluated for changes in electrical resistivity, density, and microstructure.
TITLE:
Measurement of $^{27}\text{Al}(n,2n)^{26}\text{Al}$ Cross Section Near Threshold

INVESTIGATORS:
R. K. Smither and L. R. Greenwood

AFFILIATION:
Argonne National Laboratory

SUMMARY:
The objective of this work is to measure the $^{27}\text{Al}(n,2n)^{26}\text{Al}$ reaction cross section near threshold.

Thirty-seven samples of high-purity aluminum were irradiated at the RTNS-II facility at LLNL for 2 weeks in late October and early November. The range of neutron energies seen by each sample was controlled by placing the sample at an appropriate distance from the target and at the appropriate angular position relative to the incoming deuterium beam direction. This work covered a range of neutron energies from 14.1 to 14.7 MeV. Dosimetry foils were placed adjacent to the Al samples to monitor the integral neutron flux. The maximum integrated flux observed for the closest sample (1.35 cm) was approximately $3 \times 10^{17} \text{n/cm}^2$. After irradiation the Al samples and the dosimetry foils were shipped to Argonne National Laboratory (ANL), where the activity measurements were made for the Fe and Ni reactions in the dosimetry foils and the Al $^{(n,a)}$ reaction in the Al samples. After an appropriate cooling period (3 weeks) the measurement of the long-lived activity from the decay of the 5+ ground state (g.s.) of $^{26}\text{Al}$, a 1.8-MeV gamma ray, was begun.

The counting of the long-lived activity and the subsequent calculation of the $(n,2n)$ cross sections is still in progress, but the preliminary results suggest that the cross section decreases to a near zero value at the threshold for the direct production of the first 3+ state at 14.0 MeV rather than at the threshold of the 5+ g.s. at 13.5 MeV. This confirms the change in effective threshold inferred by the previous ANL work (see Fig. 9).

The only previous measurements made on the $^{27}\text{Al}(n,2n)^{26}\text{Al}$ reaction cross section for the production of the 5+ g.s. were made at ANL. These
Fig. 9. Comparison of the experimentally determined cross sections for the productions of the $^{26}$Al ground state (730,000 years, filled circles) and the isomeric state (6 s, filled squares) with the predicted neutron energy spectra from a fusion reactor plasma at ion temperatures of 1 and 15 keV (dashed lines) and the addition of a 120-keV neutral beam injected into the plasma (dotted line).
measurements used the accelerator mass spectrometer (AMS) system at the Argonne Tandem to measure the production rate of the $^{26}$Al g. s. in a set of small Al foils irradiated in a previous experiment at RTNS-II. The very long half-life, of the g. s. decay, $7.3 \times 10^5$ years, and the small size of the foils, typically 2 to 5 mg, made direct counting very difficult. These previous measurements covered the neutron energy range from 14.4 to 14.8 MeV. The new irradiations reported here overlap this energy region and extend the measurements down to 14.1 MeV. The theoretical threshold energy for the production of the 5+ g. s. is 13.54 MeV and most of the cross section near threshold would normally be associated with the direct product of this state. The Argonne AMS data strongly suggested that this was not the case and most of the near threshold cross section was associated with the direct production of the first 3+ state at 416.9 MeV. As was mentioned in the previous paragraph, the preliminary analysis of the new data supports this suggestion and moves the effective threshold for the $(n,2n)$ reaction up to 14.0 MeV.

This increase in the effective threshold energy for the production of the 5+ g. s. has two important consequences:

1. First, it drastically reduces the amount of $^{26}$Al produced by fusion plasma (D-T) neutrons (by a factor of 5 for a 9-keV plasma) and thus reduces the long-lived radioactivity associated with it by a similar factor. This considerably reduces the disposal problem for aluminum structural materials and also for Al containing blanket materials.

2. The second consequence is that this change in threshold makes the $^{27}$Al$(n,2n)^{26}$Al reaction an attractive candidate for use in a new method for measuring the ion temperature of a D-T plasma. The new threshold is now almost exactly in the middle of the neutron energy spectrum shown in the figure. This makes the yield of this reaction very sensitive to the width of the neutron energy spectrum and thus sensitive to the ion temperature. Most neutron reactions are sensitive to only the shift of the centroid of the neutron energy spectrum, which changes very little with ion temperature and is therefore very insensitive to changes in ion temperature. By measuring the ratio of the $Al(n,2n)$ yield to the $Al(n,a)$ yield in the same dosimetry sample one obtains a value for the ion temperature without having to make an absolute measurement of anything and without any information about ion
densities or any other plasma parameters. The success of this method depends on having accurate measurements of the Al(n,2n) cross sections near threshold, which is what this work is designed to produce.

PUBLICATION STATUS:

Results of the previous Argonne work have been published in the DAFS Report DOE/ER-0046/13, p. 27 (1983) and will appear in the Journal of Nuclear Materials in a special issue devoted to papers presented at the Third Topical Meeting on Fusion Reactor Materials (Albuquerque, NM, September 19-22, 1983).

The results of this work will be published in future DAFS reports and in the Journal of Nuclear Materials and/or The Physical Review.
Effect of Impurities on the Mechanical Properties of Metals Irradiated with 14-MeV Neutrons

E. R. Bradley, J. L. Brimhall, and R. H. Jones

Pacific Northwest Laboratory

The purpose of this work is to evaluate the effects of impurities on the radiation-induced embrittlement of fusion reactor materials. The effect of interstitial impurities in Nb, V, and Fe alloys is being studied by tensile tests, TEM, and Auger electron spectroscopy. This evaluation is important for establishing impurity effects in these materials and thereby aiding in selecting embrittlement resistance fusion reactor materials and establishing limits for impurity pickup during reactor operation.

Wire tensile samples and foil TEM discs of Nb (two oxygen levels) and V have been irradiated at 300, 473, and 673 K to fluence levels ranging from 1 to $2 \times 10^{21} \text{ m}^{-2}$ with T(d,n) neutrons from the RTNS-II facility. A strong effect of oxygen content on the yield strength increase in Nb was found following irradiation at 300 K (Fig. 10). The Nb wires containing 185 wt ppm oxygen showed two distinct hardening stages that were connected by a plateau region. Increasing the oxygen content to 480 wt ppm shifted the high fluence hardening stage to a lower fluence level and eliminated the plateau region. A single hardening stage was also found in V irradiated at 300 K, which was similar to that observed in Nb containing 480 wt ppm oxygen. The V wires contained 920 wt ppm oxygen, and the single stage hardening response is consistent with the Nb results.

Defect clusters produced during irradiation provide obstacles to dislocation movement and are responsible for the observed increase in yield strength. Interstitial impurities can affect the hardening response by interacting with the irradiation-produced defects and changing the
Fig. 10. Yield strength increase vs neutron fluence for niobium irradiated with T(d,n) neutrons at 300 K.
distribution and strength of the defect clusters. Irradiation at elevated temperatures is expected to change both the distribution and strength of defect clusters because of greater mobility of interstitial impurities and irradiation-produced point defects at higher temperatures. Tensile tests and TEM examinations are currently being conducted on the V and Nb samples irradiated at 473 and 673 K to determine the temperature dependence of the strength and microstructure of these materials.

Foil samples of 316 stainless steel, Ht-9, and a dilute Fe-P alloy were included in the elevated temperature irradiations to explore possible irradiation-induced grain boundary segregation. These samples will be fractured intergranularly and the grain boundary chemistry measured by Auger electron spectroscopy. These studies will complement ongoing experiments on surface segregation following heavy ion irradiation and grain boundary segregation induced by 16-MeV proton irradiations.

PUBLICATION STATUS:


Results from the elevated temperature irradiations will be published first in the DAFS Quarterly and subsequently in technical journal articles.
Title: Neutron Sensitivity of Operational Amplifiers

Investigators: F. J. Deadrick*  
R. F. Wuerker**

Affiliation:  
*Lawrence Livermore National Laboratory  
**TRW Energy Technology Division

Summary:
A selection of junction field effect (JFET) linear operational amplifiers were irradiated at fluence levels that are expected from the MFTF-B fusion test reactor to determine the degree of performance degradation caused by 14-MeV neutrons. These amplifiers will be used in an ion spectrometer that is being designed by TRW as part of the plasma diagnostic instrumentation for MFTF-B. Design constraints preclude the use of extensive neutron shielding around the spectrometer front-end electronics assembly, and survivability of operational amplifiers is a critical design issue.

Two packages of amplifiers were irradiated out-of-circuit at the RTNS-II facility on December 19, 1983. The first package consisted of the following pre-tested amplifiers: one National LF-157, LF-157A, and LF-375A, one Precision Monolithic Inc. LM-108A, and one Burr Brown OPA-102BM, plus the following untested amplifiers: National LM-101, Precision LM 108A, and a Motorola MC-1714G. This package received a fluence of \(1.2 \times 10^{12} \text{n/cm}^2\) at 155 cm from the source.

The second package consisted of one each of the following untested amplifiers: National LF-157A, LF357A, LM-101, and a Precision Monolithic LF-108A. This package was placed 55 cm away from the source where it received \(1.4 \times 10^{13} \text{n/cm}^2\).

To date, two of the amplifier types that were irradiated have been tested. The test arrangement used both before and after irradiation is shown in Fig. 11. In addition to monitoring the response of the amplifiers to current pulses, the output noise rms voltage and offset voltages were also measured.
Fig. 11. Schematic of the operational amplifier test circuit.
Output of the Burr Brown OPA-102BM amplifier to 5 nA current pulses, before and after $1.38 \times 10^{12}$ n/cm$^2$ irradiation, is shown in Fig. 12. For this amplifier, no effort was made to correct the amplifier offset voltage by its own internal trimming circuit. The before and after noise offset output voltages as well as supply current are summarized in Table 3. It was found that the noise voltage decreased slightly, while the output offset voltage essentially doubled. Had the offset trim circuit been used, the offset could have been corrected to zero in both cases.

A similar set of pictures for the LF-357A is presented in Fig. 13. The pictures for zero and $1.15 \times 10^{12}$ n/cm$^2$ are for the same amplifier. The picture for $1.4 \times 10^{13}$ n/cm$^2$ is for an amplifier that had not been tested prior to irradiation. Measurements for rms output noise voltage and offset voltages before and after irradiations are also shown in Table 1. This amplifier had a circuit for zeroing its offset voltage, which was used. After irradiation to $10^{12}$ n/cm$^2$ the offset could still be adjusted to zero. However, the amplifier that received $10^{13}$ n/cm$^2$ could only be reset to a minimum of 0.2 V of output offset. This amplifier, while definitely "sick," still responded to the "standard" 5 nA pulse, in spite of its 0.2 V offset. The rms noise doubled. The 357-A amplifier that was irradiated to $10^{12}$ n/cm$^2$ behaved similarly to the Burr Brown OPA-102.

The present tests show that the JFET-bipolar operational amplifiers can be expected to tolerate at least $1.4 \times 10^{12}$ n/cm$^2$. For MFTF-B fluxes of $5 \times 10^7$ n/cm$^2$/s and 3000 s of machine operation per week, the operational amplifiers would last for at least 9 weeks of machine time, at which time they would be replaced.

**PUBLICATION STATUS:**

The results from these initial irradiation tests have not been published, pending a more extensive series of irradiations that will be performed during 1984. Further details may be obtained by contacting the investigators.
Fig. 12. Responses of OPA 102 BM 1 mV/nA current to voltage amplifier to 5 nA input current pulses. Recorded with oscilloscope amplifier with 1-MHz bandwidth.
<table>
<thead>
<tr>
<th>Amplifier</th>
<th>Fluence (n/cm$^2$)</th>
<th>Cutoff noise true rms (mV)</th>
<th>Output offset voltage (uV)</th>
<th>Supply (mA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Burr Brown</td>
<td>0</td>
<td>0.18</td>
<td>0.145</td>
<td>4.3</td>
</tr>
<tr>
<td>OPA-102 BM</td>
<td>--</td>
<td>0.387</td>
<td>0.23</td>
<td>3.6</td>
</tr>
<tr>
<td>National</td>
<td>0</td>
<td>0.54</td>
<td>0$^a$</td>
<td>3.8</td>
</tr>
<tr>
<td>LF-357A</td>
<td>$1.1 \times 10^{12}$</td>
<td>0.49</td>
<td>0$^a$</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$1.4 \times 10^{13}$</td>
<td>1.04</td>
<td>200</td>
<td></td>
</tr>
</tbody>
</table>

$^a$ Output adjusted to zero.
Prior to irradiation

After irradiation to $1.1 \times 10^{12}$ n/cm$^2$

Different LF357A after $1.4 \times 10^{13}$ n/cm$^2$ irradiation

Fig. 13. Responses of LF 357A 1-mV/nA current to voltage amplifiers to 5 nA input current pulses, recorded with oscilloscope amplifier with 1-MHz bandwidth.
TITLE:
Low-Dose Fission Neutron-Induced Swelling of MgAl\textsubscript{2}O\textsubscript{4} Spinel--Part of a Fission/Fusion Neutron Correlation Study

INVESTIGATOR:
F. W. Clinard, Jr.

AFFILIATION:
Los Alamos National Laboratory

SUMMARY:
It is of utmost importance to establish the correlation between bulk damage to ceramics from fission and 14-MeV neutrons, since the former are currently used for high-dose studies but the latter are a major component of a fusion reactor irradiation environment. To attain such a correlation, a collaborative experiment has been initiated between Japanese and U.S. scientists to compare swelling response of MgAl\textsubscript{2}O\textsubscript{4} irradiated to low doses with fission and fusion neutrons near room temperature. The Japanese investigators (N. Itoh, Nagoya University and C. Kinoshita, Kyushu University) are carrying out the 14-MeV neutron irradiations at RTNS-II, while the U.S. investigator (F. Clinard) is responsible for the fission neutron study.

The latter work will be carried out at the Omega West reactor at LANL, where the desired dose of roughly $10^{21}$ n/m$^2$ can easily be attained. To date, a comparitor-based technique has been developed for measurement of the extremely low strains expected (about $10^{-5}$), and precision samples have been prepared. Irradiations are planned for 1984.

PUBLICATION STATUS:
The fission neutron study is described by F. Clinard in Proc. of the RTNS-II Experimenters Workshop (Osaka, Japan, October 1983).
TITLE:
RTNS-II Irradiation of Ceramic Insulated Instrument Components in Support of FMIT Test-End Design

INVESTIGATOR:
C. P. Cannon

AFFILIATION:
Hanford Engineering Development Laboratory

SUMMARY:
Irradiations were conducted on ceramic-to-metal seals and thermocouple wire types in May, 1983 to create a technical basis from which to design instrument components for the Fusion Materials Irradiation Test (FMIT) test-end region. Five ceramic-to-metal seal types were irradiated to a fluence of $2 \times 10^{18}$ n/cm$^2$ in RTNS-II. One seal type lost hermeticity at that fluence; the other four types, including the type installed in the FMIT acceleratory stage constructed at LANL, remained hermetic to 10$^{-9}$ cc He/s.

Results to date suggest that a tensile bond configuration will not remain hermetic in a 14-MeV neutron fluence. Post-irradiation evaluation of this experiment is still in progress.

The FMIT accelerator will require ceramic-to-metal seals that remain hermetic in the 14-MeV fusion neutron fluence to $10^{21}$ n/cm$^2$. There is no practical way to conduct prototypic irradiation evaluations to that fluence. The intent of the present effort is to establish a technical basis for seal design from lower fluence 14-MeV data and high fluence fast-fission neutron data.

Fast-fission neutrons can cause loss of hermeticity in ceramic-to-metal seals at fluences as low as $10^{17}$ n/cm$^2$—the effects are neutron energy dependent. Parameters that affect the performance of seals are: configuration (geometry); ceramic type, purity, and grain size; metallization material and process; metal sleeve material and configuration; and braze type. This present RTNS-II irradiation evaluated the effects of alumina purity and grain size, and seal configuration.
Figure 14 shows the ceramic seal and thermoelement components irradiated in the present experiment. Additional experiments may be required to resolve remaining questions on ceramic-to-seal design. However, data on thermoelement wires should complete the data base from which to design thermocouples.

PUBLICATION STATUS:

Results of this experiment are scheduled to be issued in an informal report to the FMIT project by May 1984.
Fig. 14. Ceramic-to-metal seal components and thermoelement wires along with the irradiation test fixture—irradiated to $2 \times 10^{18}$ n/cm$^2$, 14-MeV at RTNS-II in May 1983.
Cryogenic (4.2 K) Irradiation of Superconductors and Stabilizers

INVESTIGATORS:
M. V. Guinan, R. A. Van Konynenburg, J. B. Mitchell*
T. Okada**
K. Suzuki***

AFFILIATIONS:
*Lawrence Livermore National Laboratory
**Osaka University
***Tohoku University

SUMMARY:
Seven samples were irradiated at the same time, including one Nb$_3$Sn "bronze process" monofilament and two Nb$_2$Sn "in situ process" samples, as well as two Mo-Si amorphous superconductors and two Cu wires. They were mounted side by side on the cold finger of a continuous flow cryostat. The Nb$_3$Sn bronze process monofilament sample was supplied by LLNL using material from Brookhaven National Laboratory. The Nb$_2$Sn in situ process wires were supplied by T. Okada from material made by K. Tasohama of Nihon University. The Mo-Si amorphous samples were supplied by K. Suzuki of Tohoku University. The Cu wires were supplied by LLNL using material from Oak Ridge National Laboratory.

The samples were repeatedly irradiated in five legs at 4 to 5 K with 14.8-MeV neutrons, up to a total fluence of about 1.35 x 10$^{22}$ n/m$^2$. After each leg, measurements of the critical current of the superconductors and the magnetoresistance of the other samples were carried out at transverse fields up to 12 T. The critical temperatures of the superconductors were measured before and after the entire series of irradiations. Although considerable effort was directed toward keeping the samples near liquid helium temperature over the entire course of the several-week-long experiment, production of gas caused by (n, alpha) reactions and radiolysis of the epoxy adhesive as well as entry of particles of solid air and/or ice into the liquid helium system during refilling caused temperature excursions to 50, 38, 40, 29.5, 62.5, and 61.5 K during or after the second through fifth irradiation leg. However, based upon the work of others, we do not believe that significant annealing of irradiation-produced defects occurs in Nb$_3$Sn at these temperatures.
After five cycles of irradiation and measurement the cryostat would no
longer operate at 4 K and had to be warmed to room temperature. A broken
current lead on one of the in situ Nb₃Sn samples was then repaired, the ice
and/or solid air were allowed to evaporate, and the cryostat was recooled to
4.2 K. A final series of measurements was then made on critical current and
magnetoresistance, and the critical temperatures were also remeasured.

The result of the irradiation up to a fluence of 1.35 x 10¹² n/m² at 4 K
on the Nb₃Sn monofilament was a monotonic, saturating increase in the critical
current as a function of fluence, in general agreement with past irradiations
of non-optimized Nb₃Sn. At 12 T, the critical current increased to three
times its original value. At 4 T, the increase was by a factor of two. After
room temperature annealing, partial recovery of the critical current increase
was observed. At 12 T, the recovery amounted to 16% of the irradiation-induced
change. At 4 T, it was 9.8%.

The critical temperature of the monofilament Nb₃Sn was observed to drop
from 17.28 K before irradiation to 16.00 K after the full fluence. The
measured value after room temperature annealing was 15.99 K.

This experiment reached the highest fusion neutron fluence yet achieved
on cryogenic samples, and probably represents the practical maximum for the
RTNS-II output available at present and the current cryostat system. The
experiment required monitoring around the clock for more than three months and
used over 10,000 liters of liquid helium. Unfortunately we were still unable
to bring about the expected final collapse of the critical current, and this
remains a goal for the future. With the incorporation of larger targets on
RTNS-II and the increase in accelerator current, it is anticipated that a
significantly higher neutron output will be available in the future.

PUBLICATION STATUS:

A preliminary report comparing results for the "bronze process"
monofilament to other studies of identical samples has been submitted to the
Sixth Annual Progress Report on Special Purpose Materials for Magnetically
Confined Fusion Reactors. Results for all the samples will be presented in
several joint papers at the First International Conference on Fusion Reactor
Materials to be held in Tokyo in December, 1984.
TITLE:
Room Temperature Irradiation of A15 Superconductors with 14-MeV Neutrons

INVESTIGATORS:
C. L. Snead, Jr.*
M. W. Guinan**

AFFILIATION:
*Brookhaven National Laboratory
**Lawrence Livermore National Laboratory

SUMMARY:
Samples of Nb₃Sn (in both core and monofilament) V₃Ga, NbSn(Ga), and NbTi reached a final dose of 5.4 × 10¹⁸ n/cm² in an add-on irradiation in the summer of 1983. Irradiation of these samples began in 1975 at the RTNS-I. After each fluence increment of 1 × 10¹⁸ n/cm² they were returned for measurement of critical properties up to fields of 20 T at the National Magnet Laboratory of the Massachusetts Institute of Technology. Results to a fluence of 3 × 10¹⁸ n/cm² have already been reported. At this fluence all the A15 superconductors showed significant degradation in critical currents. The irradiations were continued to the higher fluence of 5 × 10¹⁸ n/cm² to facilitate comparisons with fission reactor irradiations of identical samples.

PUBLICATION STATUS:
Results to 3 × 10¹⁸ n/cm² were reported at the Seattle Fusion Reactor Materials Meeting: C. L. Snead, Jr., D. M. Parkin, and M. W. Guinan, J. Nucl. Mater., 108-9, 749 (1981). Results at the final dose of 5 × 10¹⁸ n/cm² will be given at the First International Conference on Fusion Reactor Materials in Tokyo in December 1984.
Neutron Damage in High-Field Type-II Superconductors

C. L. Snead, Jr.*
M. W. Chinnan**
D. M. Parkin***

Brookhaven National Laboratory
Lawrence Livermore National Laboratory
Los Alamos National Laboratory

In 1973 a long-term series of irradiations were begun on filamentary wire specimens of A15 conductors (Nb$_3$Sn and V$_3$Ga) and NbTi at RTNS-I and at the Brookhaven High Flux Beam Reactor (HFBR). The purpose of these irradiations were twofold: (1) to establish the changes of the critical properties of the superconductors as a function of fluence for the two types of neutron irradiation and (2) by comparing the changes of the critical-properties caused by the two irradiations, to experimentally determine the damage energy of the 14-MeV neutrons. In FY 1983 an accumulated dose of $5 \times 10^{18} \text{n/cm}^2$ (14 MeV) on some of the specimens, and $4 \times 10^{18} \text{n/cm}^2$ on the remainder was achieved.

The measurements performed on these wires are critical temperature ($T_c$) and critical current ($I_c$); the latter were carried out at the Francis Bitter National Magnet Laboratory. From the $I_c$ measurements, the upper critical field $H_{c2}$ can also be obtained.

Results to date indicate that for Nb$_3$Sn the damage energy cross section for 14.8-MeV neutrons is 247 b·keV (compared with HFBR spectrum-averaged neutrons $E > 0.11$ MeV of 43 b·keV). This was based on a 14.8-MeV fluence of $3 \times 10^{18} \text{n/cm}^2$. The changes in both critical current and critical temperature were shown to scale with damage energy (fluence). For low fluences, it has been shown that $I_c$ initially increases with fluence whereas $T_c$ monotonically decreases. If one defines a critical dose as that fluence
where the $I_c$ begins to decrease (as measured at 10 T), a value of 0.19 eV/atom damage energy, or 0.0019 dpa is obtained.

Measurements on the highest fluence set is under way. The $5 \times 10^{18}$ n/cm$^2$ fluence will conclude this sequence.

PUBLICATION STATUS:

The results quoted above appeared in *J. Nucl. Mater.*, 103/104, 749-54 (1981). A composite paper detailing all the results will be forthcoming after concluding the measurements now in progress.
TITLE:
Investigation of Precipitation in Nb-Ti Alloys Using Transmission Electron Microscopy

INVESTIGATOR:
M. I. Buckett

AFFILIATION:
University of Wisconsin Applied Superconductivity Center

SUMMARY:
As a Department of Energy-Magnetic Fusion Energy Technology fellowship recipient, I completed the required 12-week practicum this past summer (May 18 to August 15) at LLNL. My interests were in the metallurgy/material science of superconducting materials. Facilities were made available at RTNS-II, where I was allowed unlimited use of the JEOL-200CX transmission microscope and thin foil preparation equipment owned by the Japanese government. I also had access to the Magnetic Fusion Energy computer and photographic labs.

My work at RTNS-II focused on observing the changes in precipitation phenomena in Nb-Ti alloys when additions of Ta or Zr are made. Its purpose was to complete some of my research toward the fulfillment of a master's degree in metallurgical engineering.

My source of support is the DOE, Magnetic Fusion Energy Technology Fellowship Program.

PUBLICATION STATUS:
The results of my work at LLNL will be included in my M. S. thesis.
TITLE:
The Aluminum-Beryllium System: The Effects of Rapid Solidification on Microstructure, Phase Relations and Phase Transformations

INVESTIGATOR:
C. M. Tanner

AFFILIATION:
Lawrence Livermore National Laboratory

SUMMARY:
This investigation is part of an overall program concerned with development a fundamental understanding of the modes and morphologies of rapidly solidifying alloys, the nature and relationships of the resulting stable and metastable phases, and the character of subsequent solid-state transformations within these rapidly solidified structures. The Al-Be system was chosen as a model system because it is a simple eutectic between the primary elements, and there are no intermediate phases or compounds present. Alloys of 0.5 to 35.0 at.% Be were liquid-quenched by arc-hammer splatting and ribbon-spinning in an inert atmosphere to produce a wide variety of fine-scaled metastable microstructures. Specimens were examined in Monbusho’s JOEL 200CX electron microscope at RTNS-II, and various types of segregated and non-segregated microstructures were identified. In addition, a new metastable G. P. Zone phase of fcc crystal structure was discovered to form from the solid state. These observations are being analyzed in light of current theories of solidification and phase transformations.

PUBLICATION STATUS:
Papers are being prepared for presentation at the 5th International Conference on Rapidly Solidified Metals (Germany, September 1984).
TITLE:
Production of Noble Gases by 14-MeV Neutrons

INVESTIGATORS:
R. J. Borg, D. A. Leich, and V. B. Lanier

AFFILIATION:
Lawrence Livermore National Laboratory

SUMMARY:
We started an investigation into the production and mechanisms of loss of noble gases from meteorites. Neutrons with an energy of 14.2 MeV generated by RTNS-II were used to induce nuclear reactions in a selection of naturally occurring minerals and pure chemical compounds. The amounts and isotopic ratios of the resulting noble gases were determined by mass spectrometry. Measurements of outgassing rates are in progress to determine specific diffusion coefficients.

PUBLICATION STATUS:

J. R. Srour, Z. Shanfield, and R. A. Hartmann

Northrop Research and Technology Center

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.

Irradiations of small-geometry pinch resistors, bipolar transistors, and MOS transistors have been performed. In addition, analytical determination of the energy and angular distributions of primary knock-on atoms in 14-MeV neutron-irradiated silicon was made. These distributions were employed in a distributed cluster model that was used in an attempt to account for measured resistance changes in the irradiated pinch resistors. In the bipolar transistor experiments, electrical measurements were dominated by damage produced in the extrinsic base region. An approach was developed for determining the effect on current gain of a single interaction in the intrinsic base region, and such experimentally-based calculations are planned. In the MOS transistor experiments, observed threshold voltage shifts appeared to be largely attributable to background ionizing radiation present during the neutron irradiations. Slope changes were noted occasionally in plots of threshold voltage shift vs neutron fluence. Further study is needed.
to determine whether these slope changes are attributable to neutron interactions, to ionizing radiation, or to device instabilities.

PUBLICATION STATUS:
Results of this study appear in the final report for Contract DNA01-82-C-0055 and in a paper published in the IEEE Transactions on Nuclear Science, pp. 4526-4532 (December 1983).
Search for a Miscibility Gap in the Cu-Ni System

C. E. Violet and R. J. Borg*

R. M. Catchings**

*Lawrence Livermore National Laboratory

**Howard University

Neutron diffraction results on the Cu-Ni system strongly suggest the presence of a low-temperature miscibility gap in the Cu-Ni phase diagram, which had previously been considered to be a continuous solid solution. We use the high-energy neutrons to produce vacancies that will enhance the diffusion of Cu and Ni at ambient temperatures toward the two-phase region. The ac susceptibility of these alloys, which have been quenched, annealed, and irradiated, will be measured to determine whether magnetic effects are associated with the two-phase region. We also plan to irradiate TEM samples to determine whether irradiation does indeed produce a two-phase system.

Preliminary results of this work were given at the American Physical Society meeting in Detroit, MI, March 26-30, 1984.
In order to do Mossbauer spectroscopy of $^{181}$Ta it is desirable to use a $^{181}$W Mossbauer source. The possibility of making such a source by means of the $(n,2n)$ reaction on $^{182}$W was explored by exposing a 1-mil foil of natural W (at. % abundance of $^{182}$W is 26.3\%) to fluences up to $\approx 2 \times 10^{18}$ n/cm$^2$.

Following the irradiation, pulse height analysis failed to show the 6.2 keV gamma-ray Mossbauer transition in $^{181}$Ta. The next step is to use a W target enriched in $^{182}$W. This will be in the form of $^{182}$O$_3$ powder, which has already arrived from Oak Ridge National Laboratory.

If we are successful with the $^{182}$O$_3$ target, we plan to publish a formal report.
TITL E:  
Measurement of Fission Yields for $^{235}\text{U}$, $^{238}\text{U}$, and $^{239}\text{Pu}$

INVESTIGATORS:  
D. R. Nethaway and F. F. Momyer

AFFILIATION:  
Lawrence Livermore National Laboratory

SUMMARY:  
We have irradiated targets of $^{235}\text{U}$, $^{238}\text{U}$, and $^{239}\text{Pu}$ with neutrons of 14.3 to 14.8 MeV. The main purpose was to measure the fission yields of 10.7-year $^{85}\text{Kr}$, $^{156}\text{Eu}$, and $^{161}\text{Tb}$. Accurate knowledge of these yields is necessary for the interpretation of diagnostic measurements made on gas and core samples of debris from nuclear tests. The fission yield of $^{85}\text{Kr}$ had not been measured previously. The yields of $^{156}\text{Eu}$ and $^{161}\text{Tb}$ had been measured before at 14.8 MeV, but we were able to remeasure them with greater accuracy.

The targets consisted of 1 g $^{235}\text{U}$ and $^{239}\text{Pu}$ metal and 3 g $^{238}\text{U}$ oxide sealed in aluminum cans. The cans were wrapped in cadmium foil to absorb thermal neutrons and sealed in separate outer steel containers. After the irradiation we dissolved the aluminum cans with acid in a closed system and collected the gaseous products for krypton and xenon measurements. We also took aliquots of the solutions for direct gamma-ray assay and for rare-earth analyses. The number of fissions in each sample was calculated from known fission yields of the products $^{95}\text{Zr}$, $^{99}\text{Mo}$, $^{144}\text{Ce}$, and $^{147}\text{Nd}$. Table 4 summarizes the results for $^{85}\text{Kr}$, $^{156}\text{Eu}$, and $^{161}\text{Tb}$.

PUBLICATION STATUS:  
We plan to have one more irradiation of $^{238}\text{U}$ at 14.8 MeV and $^{239}\text{Pu}$ at 14.3 MeV, and then prepare a formal report with all of the results.
Table 4. Measured fission yields of $^{85}$Kr, $^{156}$Eu, and $^{161}$Tb.
(Statistical errors are about 1% and do not include decay-scheme uncertainties
or the error in the number of fissions.)

<table>
<thead>
<tr>
<th>Target</th>
<th>Neutron energy (MeV)</th>
<th>$^{85}$Kr</th>
<th>$^{156}$Eu</th>
<th>$^{161}$Tb</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{235}$U</td>
<td>14.33</td>
<td>$3.98 \times 10^{-3}$</td>
<td>$7.14 \times 10^{-4}$</td>
<td>$4.09 \times 10^{-5}$</td>
</tr>
<tr>
<td></td>
<td>14.73</td>
<td>$4.00 \times 10^{-3}$</td>
<td>$5.35 \times 10^{-4}$</td>
<td>$4.38 \times 10^{-5}$</td>
</tr>
<tr>
<td>$^{238}$U</td>
<td>14.4</td>
<td>$2.50 \times 10^{-3}$</td>
<td>$1.04 \times 10^{-3}$</td>
<td>$6.89 \times 10^{-5}$</td>
</tr>
<tr>
<td>$^{239}$Pu</td>
<td>14.8</td>
<td>$2.32 \times 10^{-3}$</td>
<td>$1.94 \times 10^{-3}$</td>
<td>$2.29 \times 10^{-4}$</td>
</tr>
</tbody>
</table>
Antireflectivity Using Etched Nuclear Tracks

In 1982, we attempted to develop gradient-index antireflectivity on fused silica using etched nuclear tracks. The application envisaged was to provide high transmission and low reflection, at wavelengths of 0.53 μm and 0.35 μm, for the focusing lenses of the Nova high power laser system under construction at LLNL. The results did not meet our requirements.

The method involved placing a 25-μm thick sheet of 92% 235U foil against the glass surface and subjecting the assembly to a thermal neutron flux in RTNS-II. The neutrons induce fission of the 235U, and the resulting fission fragments penetrate the glass to a depth of ~20 μm, leaving a linear track of damaged glass. The track is more susceptible to chemical attack than the undamaged glass. Etching an isolated track leaves a pit of approximately conical shape. A surface with a large area density of small etched tracks has a varying porosity and, therefore, a graded refractive index. If the index profile is smooth, the specular reflectivity can be very low for λ < 2d, where d is the thickness of the transition layer. For high transmission, the tracks must be etched deeply enough to meet this criterion and yet have a small enough cross section that scattering is small. These conflicting requirements imply that the cone angle of the etched track be small.

A theoretical analysis showed that, under the assumptions of perfectly conical pits and random incidence of the fission fragments, the refractive index profile should be sufficiently smooth to provide good broadband antireflectivity. The analysis also showed that, for practical purposes, considerable variations of fission fragment fluence could be compensated by varying the etching depth. Deep etching, however, increases the surface scatter.
After considerable experimentation with etching, aimed at minimizing the cone angle, a standard etchant known as "P-etch" (3 HF - 2 HNO₃ - 60 H₂O, by volume) was selected. Examination of surfaces by scanning electron microscopy indicated that the resulting cone angles were about 25 deg. The apices and ridges between pits were slightly rounded, with the rounding affecting a larger portion of smaller pits.

The antireflectivity achieved was broadband but modest. In the best case (~1540 etched tracks/µm²) for fused silica treated on both surfaces, transmission at normal incidence was 95.5% at both 351 and 528 nm; the corresponding spectral reflectances were 1.3 and 2.1%. For untreated bare fused silica, the transmission at both wavelengths is about 93%. Based on scanning electron microscopy of the etching of isolated tracks, the etching depth was adequate so we attribute the remaining surface reflectance to apex and ridge rounding, which causes large gradients in the index profile. The remainder of the transmission losses are caused by diffuse surface scatter, since absorptive losses are negligible. The scattering losses increased with etching depth. The spectral reflectance of the glass surfaces could be reduced to very low values simply by increasing the scattering, but the total transmission was reduced (in one case, T = 90.0% and R = 0.4% at 351 nm). The scattering losses were found to be proportional to λ⁻ⁿ. For heavily etched surfaces (large pits), n = 4.0 as expected from standard theories. For more lightly etched surfaces n was larger, with values as high as n = 14 being observed. High values of n are not understood theoretically.

The residual radioactivity (β - γ) could be described for times between ~1 and ~160 days, by

\[
\text{activity (mR/hr) = 55.0 x [etched track density (µm⁻²)]} \times \text{time in days}^{-1.146}.
\]

Comparison with the work of Stevens and Cody,² who used an accelerator to produce damage tracks, suggests that normal incidence of the tracking particles provides some advantage. This study is now complete.

REFERENCES:

PUBLICATION STATUS:

An article entitled "Broadband Antireflectivity on Silica Using Etched Nuclear Tracks," by Robert W. Hopper, has been prepared for submission to the Journal of Applied Optics.
TITLE:
14-MeV (n,2n) Neutron Cross Sections of $^{241}$Am and $^{243}$Am

INVESTIGATORS:

AFFILIATION:
Los Alamos National Laboratory

SUMMARY:
The purpose of this experiment was to measure the $^{241}$Am(n,2n)$^{240}$Am and the $^{243}$Am(n,2n)$^{242}$Am cross sections at 14.9 MeV. Irradiations were conducted at the RTNS-II Facility.

The target materials consisted of chemically and isotopically pure $^{241}$Am and $^{243}$Am. Sample preparation was identical for both samples. Each target consisted of ~125 µg of either $^{241}$Am or $^{243}$Am and a flux monitor of ~150 µg of Tm. The Am and Tm were dissolved in 2M HNO$_3$ and evaporated to dryness on fine aluminum powder. The Al powder was cold pressed into pellets 6.4 mm in diam and 1 mm thick and coated with gold. Each pellet weighed ~30 mg. The target holder was a 38-mm-diam Al cylinder with two separate 0.025 mm Al windows that provided double containment for the Am. Two Tm metal foil flux monitors were irradiated with each pellet. In order to prevent backscatter reactions the pellet was separated from the Tm foils by 0.025 mm Al foils.

The targets were irradiated at the RTNS-II for a period of 24 hours to provide a total neutron fluence of ~10E17 n. Because of the short half-life of the $^{240}$Am, the $^{241}$Am target was opened 24 hours after the irradiation in the RTNS-II hot cell, and the pellet was shipped to LANL for analysis. The $^{243}$Am target was allowed to cool for nine days before shipment.

Each sample was dissolved in 2 ml of HCl containing 2 drops of HNO$_3$. The sample was metathesized with NaOH to remove the excess Al. Next, the hydroxide precipitate was dissolved in HCl. The Am-Tm separation was accomplished by two passes thru a cation column that had been conditioned
with a mixture of ethanol and HCl that had been saturated with hydrogen chloride gas. Aliquots were taken for radiochemical analysis. $^{241}$Am, $^{243}$Am, and $^{242}$Cm (the decay product of $^{242}$Am) were measured by alpha pulse analysis. The gamma activity from the $^{240}$Am was measured in a NaI counter. The Tm concentrations were measured by standard radiochemical techniques and by neutron activation analysis.

The $^{243}$Am concentration of the solutions was also verified by isotope dilution mass spectrometry. Aliquots from each irradiation were dissolved in a mixture of methanol and nitric acid and loaded onto an anion column. The Am was eluted with a mixture of methanol and hydrochloric acid. The sample was evaporated to dryness and electroplated onto a rhenium filament. Once the Am had been plated a platinum overcoating was deposited. Each sample was analyzed at 1450 °C by thermal ionization mass spectrometry operating in the pulse counting mode.

The cross sections at 14.9 MeV are 241.4 mb for $^{241}$Am(n,2n)$^{240}$Am and 152.7 mb for $^{243}$Am(n,2n)$^{242}$Am.

PUBLICATION STATUS:

No publication is planned at this time.
TITLE:
Spallation and 14 MeV-Neutron Irradiation of Stabilized NbTi Superconductors

INVESTIGATORS:
P. Hahn*
H. W. Weber and B. S. Brown**
M. W. Guinan***

AFFILIATIONS:
*Atominstitut der Oesterreichischen Universitaeten-Wein, Vienna, Austria
**Argonne National Laboratory
***Lawrence Livermore National Laboratory

SUMMARY:
Irradiation of a broad spectrum of stabilized single core and multifilamentary NbTi superconductors has been carried out at room temperature to a fluence of $4-5 \times 10^{17}$ n/cm$^2$. The samples were commercial materials of different Ti concentrations subjected to different thermomechanical treatments. Following irradiations the samples were returned to all for critical current measurements. The results of the RTNS-II irradiations will be compared to results on similar samples irradiated at the Argonne Intense Pulsed Neutron Source and the Triga Mark-II reactor in Vienna. The samples will be reirradiated at the RTNS-II for an additional $3 \text{ to } 4 \times 10^{17}$ n/cm$^2$ in early 1984, and again remeasured. The results should provide information on the influence of the neutron spectrum in modifying the various flux pinning mechanisms operating in these materials.

PUBLICATION STATUS:
The results will be published first in P. Hahn's doctoral dissertation for the Atom Institute in Vienna and later as a journal article.

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