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DT FUSION NEUTRON IRRADIATION OF ANL
ELECTRON MICROSCOPY SAMPLES, LLL "TRITIUM-
TRICKED" NIOBIUM, LLL Al_2O_3 , SiC, AND
 B_4C , AND AN LLL SPUTTERING EXPERIMENT

MASTER

Susan C. MacLean

February 25, 1976

Unclassified

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For most efficient use of the LLL Rotating Target Neutron Source (RTNS), samples from several experimenters were combined for the irradiation run beginning January 19, 1976.

A copper capsule, approximately 9 mm in diameter, containing electron microscopy samples was supplied by Dr. Robert L. Lyles, Jr. of Argonne National Laboratory (ANL). He requested a fluence of 10^{15} neutrons/cm² at the front of the capsule and $1-5 \times 10^{14}$ neutrons/cm² at the back end of the samples. (About 33 mm along the capsule, it was scribed to indicate the end of the sample stack). Two 0.127 mm thick niobium dosimetry foils were attached to the capsule. One foil, cut to approximately 9 mm in diameter, was taped on the front of the capsule. The second foil was rectangular in shape (about 18 x 10 mm) except it had half a circle cut out of one of its longer sides so that it would fit around the capsule and could be taped in place at the scribed line, perpendicular to the capsule.

The copper capsule was taped parallel to a 9.5 mm diameter rod and placed at a 35° angle to the neutron source so that it would fit with

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the other experimental apparatus. In order to get the range of fluences desired along the length of the capsule, it was irradiated for only one day.

Dr. Willis L. Barmore of LLL provided five niobium samples. They were approximately 0.025 mm thick. Three of the samples had had the "tritium trick" performed on them to varying degrees. One sample each had 25, 50, and 100 ppm helium concentration. The two remaining samples had been annealed only.

The niobium samples were cut to approximate 12 mm diameter discs. The two foils which were annealed only were at the outsides of the stack of samples. The other foils (as described by their helium concentration) were arranged, from front to back, 50 ppm, 25 ppm, and 100 ppm. The stack of samples was taped into a packet and then centered on the front of the LLL sputtering experiment capsule.

Dr. Ralph H. Condit of LLL requested irradiation of three crystal samples. The first, SiC, 120 grit, was wrapped in aluminum foil, as was the second sample, B₄C. The third sample was an Al₂O₃ crystal. Each sample was sandwiched between two niobium dosimetry discs, 12 mm in diameter and 0.127 mm thick. These sample packets were taped around the top front edge of the LLL sputtering capsule. Therefore, the neutrons hit the narrow dimension of the crystals.

The LLL sputtering experiment was designed by Dr. Robert Meisenheimer. His capsule contained five niobium dosimetry foils, all measuring approximately 25 mm in diameter and 0.14 mm in thickness.

The order of stacking, beginning with the material nearest the neutron source, was as follows:

<u>Order</u>	<u>Sample</u>
1.	Copper foil
2.	Nb-F-B
3.	Nb-50-B
4.	Nb-25-B
5.	Nb-100-B
6.	Nb-R-B
7.	Nb-M-9
8.	Nb-M-10
9.	Nb-M-11
10.	Nb-M-12
11.	Nb-M-13

Around the top of the sputtering capsule, side by side

12.	Nb-548
13.	SiC in aluminum foil
14.	Nb-549
15.	Nb-553
16.	Al ₂ O ₃
17.	Nb-552
18.	Nb-550
19.	B ₄ C in aluminum foil
20.	Nb-551

Second rod, 35° angle, approximately 33 mm from neutron source

21.	Nb-554
22.	Copper Capsule
23.	Nb-555

An 0.05 mm thick copper foil with a 6 mm diameter hole in it was centered on the front of the sample stack and sputtering capsule. An autoradiograph of the copper foil after a few minutes of irradiation,

was used to center the samples with the center of the neutron beam. The irradiation was carried out during the period of January 19-23, 1976 by the LLL E Division Accelerator Staff. The neutron production was monitored continuously with a proton recoil counter and recorded every hour. The dose record is attached. The total beam-on time was 74.13 hours.

The ANL copper capsule was removed after one day of irradiation and shipped back to ANL. Following the entire irradiation, the sputtering capsule and other samples were stored for a few days to allow for decay of short-lived isotopes. The SiC, B₄C, and Al₂O₃ crystals were returned to R. Condit. The sputtering capsule was unloaded by R. Meisenheimer. He delivered the niobium dosimetry foils to the author for cutting before gamma ray counting. The front sputtering dosimetry foil was autoradiographed before cutting. This showed that the foil had been centered within 10 mm of the center of the neutron beam. A disc 10 mm in diameter was cut from the center of each of the five niobium dosimetry foils that had been included in the sputtering capsule.

W. Barmore's niobium samples were sent, along with the other niobium dosimetry foils, for gamma ray counting before they were returned to him. Ruth Anderson, in the LLL Radiochemistry Division, carried out the gamma ray counting.

The average fluence of each foil was calculated using the method described in UCRL-51393, Rev. 1. However, the cross section used for the activation of the 10.16 day isomer of niobium by 14.8 MeV neutrons was changed to 458 millibarns. The results were as follows:

<u>Dosimetry Foil</u>	<u>Fluence (neutrons/cm²)</u>
Nb-F-B	1.73×10^{17}
Nb-50-B	1.71×10^{17}
Nb-25-B	1.65×10^{17}
Nb-100-B	1.61×10^{17}
Nb-R-B	1.57×10^{17}
Nb-M-9	9.31×10^{16}
Nb-M-10	4.45×10^{16}
Nb-M-11	2.55×10^{16}
Nb-M-12	1.60×10^{16}
Nb-M-13	1.13×10^{16}
Nb-548	1.17×10^{16}
Nb-549	1.02×10^{16}
Nb-553	1.52×10^{16}
Nb-552	1.25×10^{16}
Nb-550	1.70×10^{16}
Nb-551	1.47×10^{16}
Nb-554	1.22×10^{15}
Nb-555	3.04×10^{14}

The estimated overall uncertainty of these results is $\pm 7.5\%$.

The relative uncertainty between any two values is about $\pm 2\%$. The values given here represent average fluences over the volume of each dosimetry foil.

RTNS IRRADIATION OF JANUARY 19 TO 23, 1976

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