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DT FUSION NEUTRON IRRADIATION OF LLL NIOBIUM TENSILE SPECIMEN,
LASL-BNL SUPERCONDUCTOR WIRES, LLL MOLYBDENUM,
VANADIUM AND ALUMINUM, LLL GRAPHITE, ORNL
MAGNESIUM OXIDE CRYSTAL, SLL-LLL NATURAL
QUARTZ, SYNTHETIC QUARTZ AND SUPRACIL,
ANL ELECTRON MICROSCOPY SAMPLES AND
SLL FIBER OPTIC BUNDLES

UNCLASSIFIED

Susan MacLean

March 17, 1975

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For most efficient utilization of the LLL Rotating Target Neutron Source (RTNS), samples from several experimenters were combined for the irradiation run beginning February 12, 1975.

Drs. Jack B. Mitchell and Richard A. Van Konynenburg of LLL provided a niobium tensile specimen for irradiation. Two 1 mil thick niobium dosimetry foils were cut the same size and shape as the tensile specimen.

Three superconductor wires were being irradiated to a fluence of 10^{18} neutrons/cm² for Dr. Don M. Parkin of Los Alamos Scientific Laboratory (LASL) and Dr. C.L. Snead, Jr. of Brookhaven National Laboratory (BNL). The wires, 19-core multifilament Nb₃Sn wires in a bronze matrix, had been irradiated October 1-4, 1973, December 3-7, 1973, December 10-12, 1973, January 14-16, 1974, March 11-14, 1974, May 20-23, 1974, May 29-31, 1974, January 6-14, 1975, and January 21-February 4, 1975.

Dave Hosmer, Dr. J. Birch Holt and Dr. Gordon S. Smith, all of LLL, provided 14 graphite samples measuring approximately 12mm square; nine molybdenum foils (1 mil thick), eight vanadium foils (1.5 mils thick), and eight aluminum foils (1.25 mils thick) for irradiation. They intended to measure helium generation in the samples.

A magnesium oxide crystal, MgO-8, was being irradiated to a fluence near 10^{18} neutrons/cm² for Dr. Yok Chen of Oak Ridge National Laboratory. The crystal had previously been irradiated January 14-16, 1974, May 29-31, 1974, July 16-18, 1974, September 11, 1974, October 7-9, 1974, October 25, 1974, November 7-13, 1974, December 2-6, 1974, January 6-14, 1975, and January 21-February 4, 1975.

Michael Moran of LLL and Dr. Peter L. Mattern of Sandia Livermore Laboratory (SLL) provided ten quartz samples, eight supracil samples, one natural quartz sample, and one Zcut, synthetic quartz sample for irradiation. The natural quartz sample measured approximately 10 x 20 x 3mm and the Zcut, synthetic quartz was about 10 x 10 x 2mm. Varying fluences within the range of 10^{10} to 10^{16} neutrons/cm² were requested for the quartz and supracil samples. Their sizes varied but they measured approximately 2 x 3 x 10mm.

Dr. Karl L. Merkle of Argonne National Laboratory (ANL) requested irradiation of a copper cylinder measuring approximately 3/8 inch in diameter and 2 inches long. The cylinder contained samples to be examined by electron microscopy following irradiation.

Fifteen fiber optic bundles were provided for irradiation by Dr. Lawrence M. Watkins of Sandia Livermore Laboratory (SLL). The bundles were wound in concentric circles of diameters from 10-1/2 inches to about 13 inches. Nine fiber optic bundles were taped on the front of a 60 mil thick lucite disc and six bundles were taped to the back. Six niobium dosimetry discs, 5 mils thick, 12mm in diameter, were taped to the bundles (three in front, three in back) in positions corresponding to 4 o'clock, 8 o'clock, and 12 o'clock.

The samples were fastened to 13 sample holders and arranged along three aluminum rods. The first rod was in a straight line with the neutron beam and the front sample holder, A, contained six stacks of samples. The niobium tensile specimen, the superconductor wires, the molybdenum, the vanadium, the aluminum, and part of the graphite were centered with the neutron source. The niobium dosimetry foils used were 1 mil thick and, except for those on the tensile specimen and graphite samples, were 12mm diameter discs. All the dosimetry foils sandwiching the graphite samples were 1 mil thick and approximately 12mm square. A second stack of graphite samples was to the right (as seen from the neutron source) of the centered pile on the sample holder A. MgO-8 with 1 mil thick, 12mm diameter dosimetry foils was to the left of the centered stack of samples. The natural quartz, quartz Q-10 and supracil S-8 together, and the Zcut, synthetic quartz had 1 mil thick niobium dosimetry foils cut to the same size and shape as the

samples. These samples were above the centered stack in a single layer, horizontal row. Left to right as viewed from the neutron source, they were in the order listed except that S-8 was directly above Q-10.

The second sample holder, B, on the first rod, held Merkle's capsule with a one mil thick, 12mm diameter niobium dosimetry foil directly on the front of the capsule. A doughnut shaped dosimetry foil was cut from 5 mil thick niobium foil to fit snugly around the capsule. This was placed at the line on the capsule indicating the end of the samples inside. Sample holder A had a hole in it that was covered on the front with mylar. Therefore, Merkle's capsule could be held right up behind the center samples on the first holder, flush with the mylar.

The lucite supporting the fiber optic bundles was the third sample holder on the first rod.

A second rod was used for seven sample holders. Six sample holders held both supracil and quartz samples side by side on a niobium dosimetry foil. The last sample holder held supracil only. The dosimetry foils on the first four sample holders were 5 mil thick, 12mm diameter discs. The foils on the last three sample holders of the second rod were 5 mil thick niobium measuring approximately one inch square.

The third rod held three sample holders each with one quartz sample. The first sample holder (K) on this rod had two dosimetry foils, 5 mils thick and one inch square. The other two holders each had four niobium foils 5 mils thick and one inch square.

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<u>Order</u>	<u>Sample Holder</u>	<u>Sample</u>	<u>Distance from Source</u>
20.		V-2	
21.		V-3	
22.		V-4	
23.		V-5	
24.		V-6	
25.		V-7	
26.		V-8	
27.		Nb-293	
28.		Nb-290	
29.		A1-1	
30.		A1-2	
31.	A	A1-3	
32.		A1-4	
33.		A1-5	
34.		A1-6	
35.		A1-7	
36.		A1-8	
37.		Nb-291	
38.		Nb-288	
39.		Graphite H-1	
40. (Bracketed groups arranged side by side)		Graphite H-2	
41.		Graphite H-3	

<u>Order</u>	<u>Sample Holder</u>	<u>Sample</u>	<u>Distance from Source</u>
42.	A	Graphite H-4	
43.		Graphite H-5	
44.		Graphite H-6	
45.		Nb-289	
46.		Nb-304	
47.		Graphite H-7	
48.		Graphite H-8	
49.		Graphite H-9	
50.		Graphite H-10	
51.		Graphite H-11	
52.		Graphite H-12	
53.		Graphite H-13	
54.		Nb-305	
55.		Nb-306	
56.		Graphite H-14	
57.		Nb-307	
58.		Nb-296	
59.		MgO-8 wrapped in aluminum foil	
60.		Nb-297	
61.		Nb-302	
62.		Natural quartz	
63.		Nb-303	

<u>Order</u>	<u>Sample Holder</u>	<u>Sample</u>	<u>Distance from Source</u>
64.	A	Nb-308	216mm
65.		S-8 and Q-10, side by side	
66.		Nb-309	
67.		Nb-300	
68.		Z cut, synthetic quartz	
69.		Nb-301	
70.	B	Nb-310	
71.		Merkle's capsule	
72.		Nb-331	
73.	C	Nb-311 (12 o'clock position)	
74.		Nb-328 (4 o'clock position)	
75.		Nb-313 (8 o'clock position)	
76.		Fiber optic bundles	
77.		Nb-312 (12 o'clock position)	
78.		Nb-329 (4 o'clock position)	
79.		Nb-330 (8 o'clock position)	
Second rod			
80.	D	Nb-314	31mm
81.		S-7 and Q-9	
82.		Nb-318	

<u>Order</u>	<u>Sample Holder</u>	<u>Sample</u>	<u>Distance from Source</u>
83.	E	{Nb-315	68mm
84.		{S-6 and Q-8	
85.		{Nb-319	
86.	F	{Nb-316	96mm
87.		{S-5 and Q-7	
88.		{Nb-320	
89.	G	{Nb-317	216mm
90.		{S-4 and Q-6	
91.		{Nb-321	
92.	H	{S-3 and Q-5	306mm
93.		{Nb-322	
94.	I	{S-2 and Q-4	684mm
95.		{Nb-323	
96.	J	{S-1	967mm
97.		{Nb-324	
Third rod			
98.	K	{Q-3	221mm
99.		{Nb-325 a + b	
100.	L	{Q-2	698mm
101.		{Nb-326 a,b,c + d	
102.	M	{Q-1	2.31 meters
103.		{Nb-327a,b,c, +d	

The irradiation was carried out by the LLL E Division Accelerator Staff during the period February 12 to 14, 1975. The third rod, containing sample holders K, L, and M, was removed after 30 minutes of irradiation on February 12, 1975. Neutron production was monitored continuously with a proton recoil counter and recorded each hour. The dose record is attached. Total beam-on time was 45.82 hours.

The sample holder assemblies were removed and stored for a few days to allow for decay of short-lived isotopes. An autoradiograph of the front sample holder, A, indicated that the centering of the samples had been within 1mm of the center of the neutron source.

The samples were all returned to the experimenters except for the aluminum, vanadium, and molybdenum. These were stored until the samples decayed enough and they could be safely handled and tested for helium content.

The two tensile-specimen shaped dosimetry foils were cut so that only the sections corresponding to the tensile sample gauge portion were weighed. These along with the other niobium dosimetry foils and one each of the aluminum, vanadium, and molybdenum foils, were delivered to Ruth Anderson in the LLL Radiochemistry Division. She carried out the gamma ray counting of the foils. The average fluence of each niobium dosimetry foil was calculated using the method described in UCRL-51393, Rev. 1. However, the value used for the niobium cross section for activation of the 10.16 day isomer was changed to 458m barns for 14.8 Mev neutrons. The results were as follows:

Dosimetry Foil NumberFluence (neutrons/cm²)

Sample holder A:

Nb-286	2.08×10^{17}
Nb-287	1.67×10^{17}
Nb-298	1.27×10^{17}
Nb-299	1.08×10^{17}
Nb-294	1.01×10^{17}
Nb-295	9.06×10^{16}
Nb-292	8.57×10^{16}
Nb-293	7.73×10^{16}
Nb-290	7.45×10^{16}
Nb-291	6.43×10^{16}
Nb-288	5.98×10^{16}
Nb-289	4.39×10^{16}
Nb-304	2.47×10^{16}
Nb-305	2.19×10^{16}
Nb-306	2.13×10^{16}
Nb-307	1.97×10^{16}
Nb-296	1.38×10^{16}
Nb-297	1.31×10^{16}
Nb-302	1.13×10^{16}
Nb-303	1.03×10^{16}
Nb-308	1.94×10^{16}
Nb-309	1.66×10^{16}
NB-300	1.17×10^{16}
NB-301	1.08×10^{16}

Dosimetry Foil NumberFluence (neutrons/cm²)

Sample holder B:

NB-310

 4.11×10^{16}

NB-331

 1.86×10^{15}

Sample holder C:

NB-311

 8.78×10^{13}

NB-328

 8.81×10^{13}

NB-313

 8.83×10^{13}

NB-312

 7.56×10^{13}

NB-329

 7.59×10^{13}

NB-330

 7.48×10^{13}

Sample holder D:

NB-314

 3.71×10^{15}

NB-318

 3.03×10^{15}

Sample holder E:

NB-315

 8.97×10^{14}

NB-319

 7.94×10^{14}

Sample holder F:

NB-316

 4.39×10^{14}

NB-320

 4.08×10^{14}

<u>Dosimetry Foil Number</u>	<u>Fluence (neutrons/cm²)</u>
Sample holder G:	
Nb-317	8.66×10^{13}
NB-321	8.20×10^{13}
Sample holder H:	
Nb-322	3.98×10^{13}
Sample holder I:	
Nb-323	7.59×10^{12}
Sample holder J:	
Nb-324	3.63×10^{12}
Sample holder K:	
Nb-325 a + b	8.84×10^{11}
Sample holder L:	
Nb-326 a,b,c + d	8.39×10^{10}
Sample holder M:	
Nb-327 a,b,c + d	3.63×10^9

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.

The helium generated in the aluminum samples was calculated using the gamma ray count of A1-5 sample. The neutron fluence for the sample was calculated as described in UCRL-51393, Rev. 1, using values appropriate for the reaction $^{27}\text{Al}(n, \alpha)^{24}\text{Na}$. The number of helium atoms formed was obtained by multiplying the cross section for the reaction by the fluence. Thus, having the number of helium atoms (parts per billion) for A1-5, and the fluences for niobium dosimetry foils (of known thickness) at both ends of the stack of aluminum samples (of known thickness), the helium generated for each sample was calculated by interpolation. The results were as follows:

<u>Sample</u>	<u>Helium Atoms (Atomic ppb)</u>
A1-1	8.33
A1-2	8.20
A1-3	8.07
A1-4	7.93
A1-5	7.80
A1-6	7.66
A1-7	7.53
A1-8	7.40

The estimated overall uncertainty of these results is about $\pm 3\%$.

Two (n, α) reactions occur in vanadium to produce helium: $^{51}\text{V}(\text{n},\alpha)^{48}\text{Sc}$ and $^{50}\text{V}(\text{n},\alpha)^{47}\text{Sc}$. Since ^{50}V accounts for only 0.24% of the vanadium, this reaction produces an insignificant amount of helium compared to that formed from ^{51}V . Using the gamma ray count of sample V-4, the number of helium atoms formed for each vanadium sample was calculated in the same manner as described for the aluminum samples. The results were as follows:

<u>Sample</u>	<u>Helium Atoms (ppb)</u>
V-1	1.49
V-2	1.47
V-3	1.46
V-4	1.44
V-5	1.42
V-6	1.40
V-7	1.39
V-8	1.37

The estimated overall uncertainty of these results is about $\pm 3\%$.

RTNS IRRADIATION OF FEBRUARY 12 TO 14, 1975

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