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Melt Wire Analyses for the Colorado School of Mines (CSM 16-10584) Irradiation in ATR

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Summary

PNNL project 74242 involves the analysis of neutron fluence monitors and melt wires irradiated in the Advanced Test Reactor (ATR) at Idaho National Laboratory in accordance with MPO 00236287 and Statement of Work No. 17370, Rev. 0, *PNNL Analysis of NSUF Flux and Melt Wire Capsules*. This report is for the Colorado School of Mines (CSM 16-10584) experiment which was conducted in position B5 of the ATR. Neutron fluence monitor results were reported in February 2021 in report *Neutron Dosimetry for the Colorado School of Mines (CM 16-10584) Irradiation in ATR.* This report presents the analyses of the melt wire capsules that were included along with the neutron fluence monitors. Each capsule was identified by the ID stamp on the bottom, then opened to assess the condition of the Pb and Zn-Al wires as well as the Bi powder. Pictures are included to illustrate the results.

Irradiation History

The CSM assembly consisted of 5 sections referred to as A, B, C, D, and E. All CSM sections were irradiated in ATR cycle 164A from June 12, 2018 to August 17, 2018, for 54 days EFPD (effective full power days) at 22.6 MW for a total of 1220.7 MWD (Megawatt days). Capsules A and E, located at the highest and lowest elevations in the assembly, were then removed and capsules B, C, and D were further irradiated in ATR cycle 164B from September 19, 2018 to January 17, 2019 for a total exposure of 117.6 EFPD at 22.6 MW for a total exposure of 2658.6 MWD.

Preparation of Neutron Fluence and Melt Wire Monitors

The preparation of the neutron fluence and melt wire monitors is documented in the report PNNL-70745, *CSM-10584 Monitor Procurement for Idaho National Laboratory,* MPO#00184726, SOW-13744, Rev. 2 sent to Craig Tyler on August 31, 2017. Small high-purity wires of Fe and Ti were encapsulated in vanadium capsules measuring 0.05" OD by about 0.39" long. The neutron fluence monitors are listed in Table 1. Melt wires were encapsulated in separate vanadium capsules containing Bi, Pb, and 95Zn-5Al alloy wires, as listed in Table 1. All vanadium capsules have identification codes stamped on the bottom. and each wire and the final sealed capsules were accurately weighed. The vanadium capsules were electron beam welded in a vacuum, helium leak tested, and subjected to additional tests and inspections as documented in our report.

The neutron fluence monitors and melt wire monitors were placed into the CSM assemblies as documented in drawings provided by INL and the position and elevation of each capsule relative to the midplane of the ATR are listed in Table 3 of that report [1]. The fabrication report listed

above includes analyses of the composition and melting points of each melt wire material, as determined by differential scanning calorimetry performed by IMR Test Labs.

Table 1. Location of the Neutron Fluence and Melt Wire Monitors in the CSM Assembly
Cells shaded in green did not melt. Cells shaded in light red appear to have melted. Cells shaded in yellow are for capsules that were not received (E1) or lost on opening (1X). Cells shaded in gray (Zn-Al) indicate that the wires could not be removed from the capsules such that the status could not be determined (see text).

Height, in	Capsule CSM-	Fluence Monitor	Bi 271.58 ^{0}C	Pb 328.51 ^{0}C	95Zn-5Al 383.73 ^{0}C	Implied Temperature
-24.3	E1	1Z	B9	7B	9E	T < 272 C
-22.9	E2	E5	9V	BA	5I	T < 272 C
-21.4	D1	I2	J1	IR	8Z	T < 272 C
-19.9	D2	1U	Y5	D2	97	272 C <T< 328 C
-2.0	C3	1Y	50	J2	1H	272 C <T< 328 C
-0.6	C4	8H	87	A9	5U	272 C <T< 328 C
0.7	C1	B1	2Z	80	8B	272 C <T< 328 C
2.0	C2	E2	01	E1	B2	272 C < T
19.8	B1	5L	1J	71	9B	272 C <T< 328 C
21.3	B2	7Y	7D	1X	17	T < 272 C
22.8	A1	87	Y8	98	7B	T < 272 C
24.2	A2	9Y	5Z	2V	8A	T < 272 C

Post-Irradiation Analyses of Melt Wire Monitors

PNNL received 35 of the 36 irradiated melt wire monitors listed in Table 1 in three 55-gallon drums. Each monitor was identified by the unique ID stamp on the bottom of each vanadium capsule, as listed in the table. The monitors were placed under a microscope and pictures were taken to document the ID number. Each capsule was then opened using tubing-cutter pliers. In cases where a wire or Bi powder was observed to fall out of the opened capsule, it is clear evidence that the wires or powder did not melt, and those cells are shaded green in Table 1. If no wire or powder was observed, additional cuts were made to see if any material could be separated from the vanadium capsule. If no wire or powder could be recovered from inside the open capsule, then it is assumed that the wire or powder melted, and those cells are colored light red in Table 1. The Pb melt wire E1 in capsule C2 was not sent to us for analysis and that cell is shaded yellow. When we opened melt wire capsule Pb1X in capsule B2, part of the capsule was lost. Hence, we were not able determine if the Pb wire melted in this case.

Pictures were taken to document the condition of each melt wire capsule. Prior to opening the capsules, we positively identified each capsule by looking at the ID stamped on the bottom using a low power microscope. Figure 1 shows some pictures of the ID stamps. We then opened each capsule to see if the Pb or ZnAl wires or Bi powder would fall out. Figures 2 to 4 show pictures of the cut capsules and any contents of every melt wire capsule to document the condition.

Figure 1 – Examples of the ID stamps used to positively identify each melt wire capsule. Left to right Bi-87, Pb-J2, Pb-1X, and ZnAl-97.

Conclusions

Table 1 summarizes our observations when the melt wires capsules were opened.

-Bi (271.58 ^{0}C)– The bismuth capsules at the top and bottom heights < -21.4" and > 21.3" do not show any evidence of melting since the powder was extracted intact. No powder was extracted from the capsules located between -19.9" and 19.8" and it is assumed that the bismuth melted at these locations.

-Pb (328.51 ^{0}C) – Lead wires were extracted from all of the capsules except for capsule BA. It is thus possible that the lead wire melted only at the location of capsule BA, and not at any other locations.

-95Zn-5Al (383.73 ^{0}C) – It was noted in the fabrication report that the 95Zn-5Al wires fit snugly in the vanadium capsules and were pushed down to avoid any melting during welding. On post-irradiation examination, none of the 95Zn-5Al wires could be extracted from the capsules except for the wire from capsule 7B. Since most of the wires were not extracted, one might conclude that all the wires melted except the one at the location of capsule 7B. However, this conclusion would be at odds with the established melting points of the two wire types, since the Pb wires, which have a melting point about 55 ^{0}C lower than that of 95Zn-5Al did not melt (except possibly at position BA). This leads us to speculate that the 95Zn-5Al wires were simply stuck in the capsules due to some effect other than melting. For example, Al generates a lot of helium during irradiation, which could lead to swelling of the nominal 0.020" diameter 95Zn-5Al wire inside the 0.031" ID vanadium capsule. Furthermore, transmutation of the Zn and Al during

irradiation may have also changed the physical properties of the alloy wire. Finally, it is possible that the 95Zn-5Al alloy wire reacted with the vanadium during irradiation at high temperature, although there is no evidence of that for wire 7B.

In summary we would suggest focusing on the behavior of the bismuth and lead wires, suggesting that the highest temperature during irradiation was less than the melting point of lead at the highest and lowest elevations (± 21") and that the temperature exceeded the melting point of bismuth but was less than the melting point of lead at core positions between -19.9 to +19.8". The behavior of the 95Zn-5Al alloy is likely anomalous and not indicative of the temperatures reached during the irradiation.

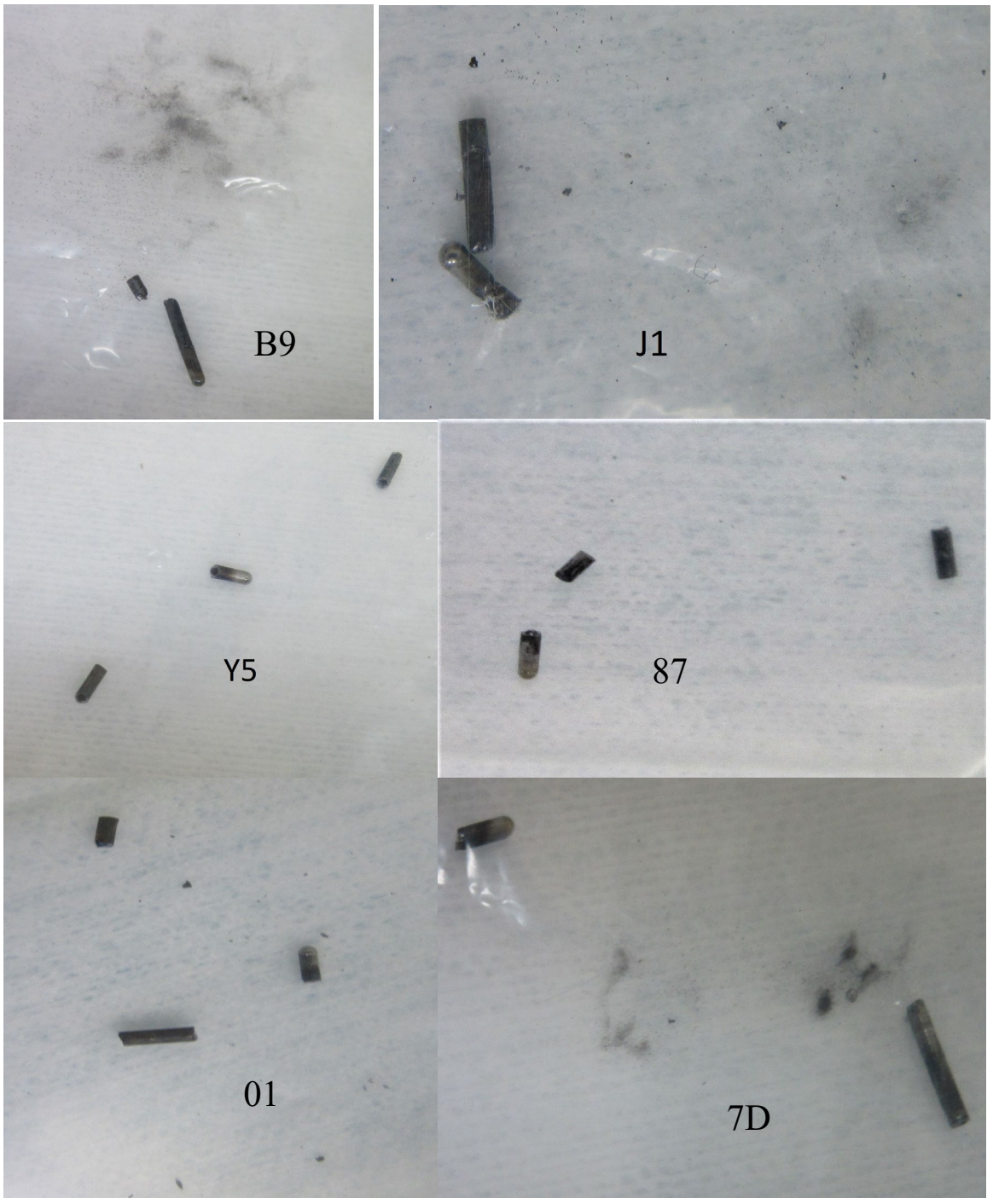

Figure 2 – Pictures (top left to bottom right) of opened Bi capsules B9, J1, Y5, 87, 01, and 7D. Capsules B9, J1, and 7D show Bi powder whereas no Bi powder could be extracted from capsules Y5, 87 and 01.

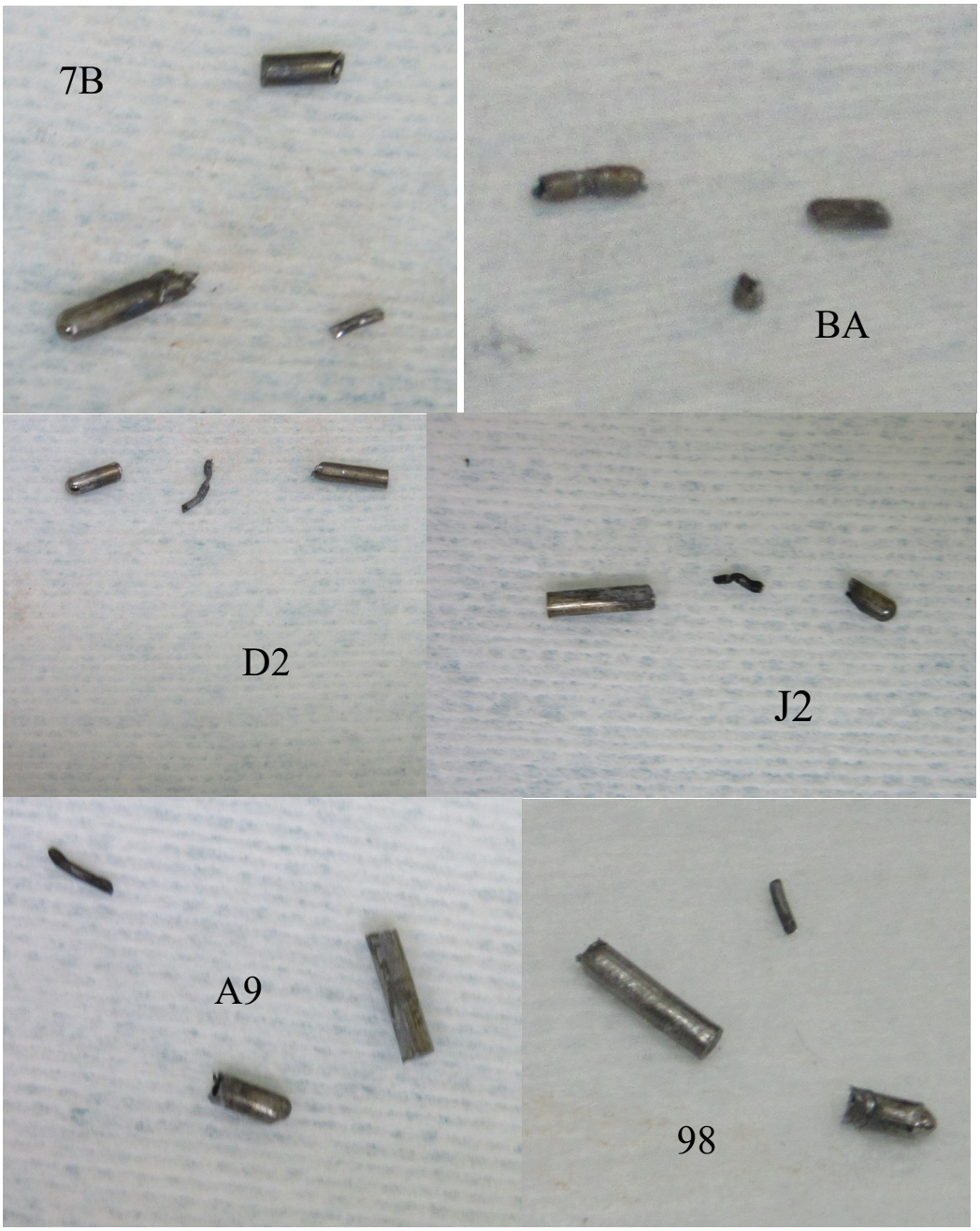

Figure 3 – Pictures of opened Pb capsules (top left to bottom right) 7B, BA, D2, J2, A9, and 98. The lead wire, seen in all pictures except for capsule BA, demonstrates that the wires did not melt.

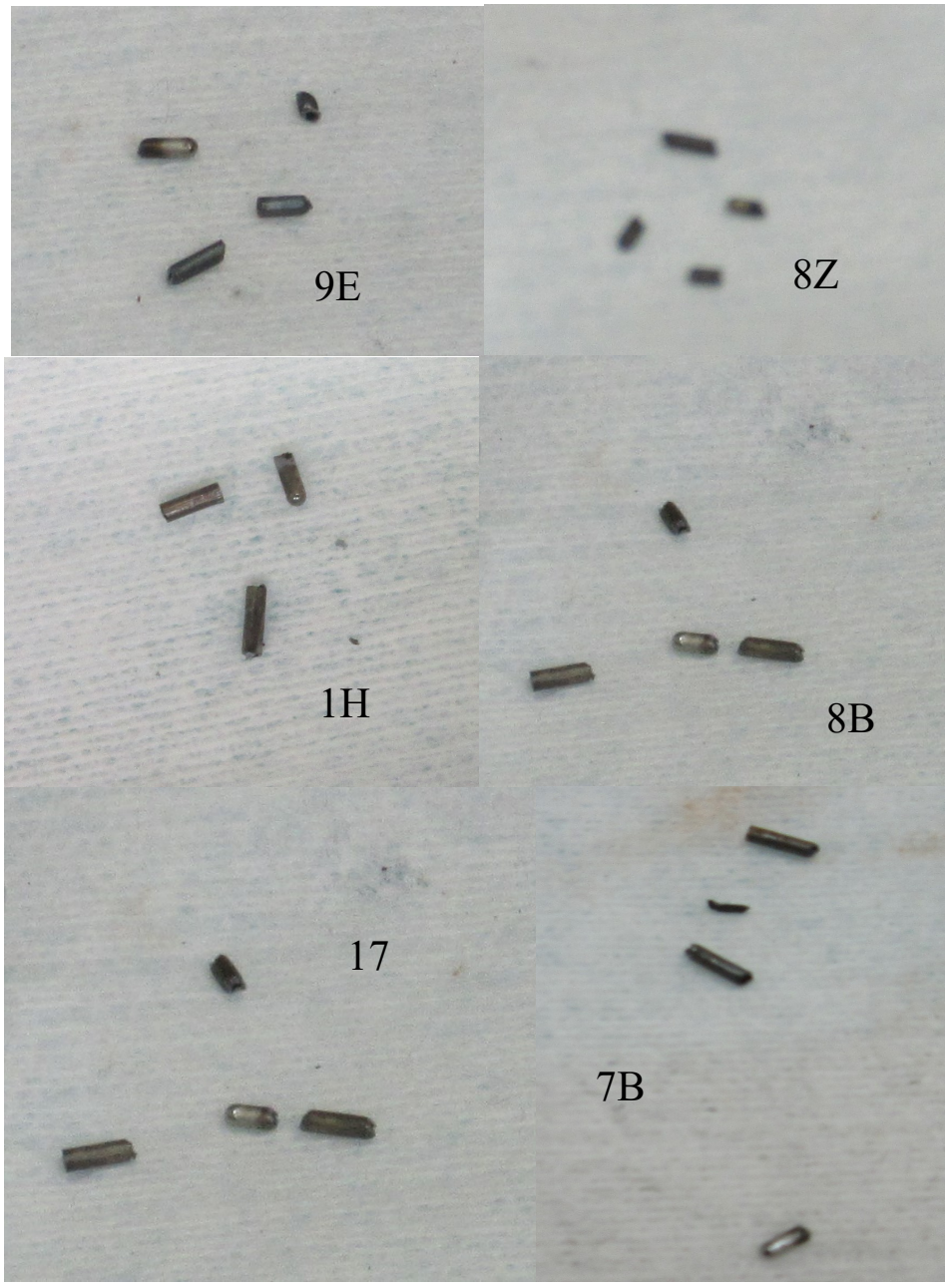

Figure 4 – Pictures of opened ZnAl capsules (top left to bottom right) 9E, 8Z, 1H, 8B, 17, and 7B. The ZnAl wire was extracted only from monitor 7B.

References

[1] L. R. Greenwood, Neutron Dosimetry for the Colorado School of Mines (CSM 16-10584) Irradiation in ATR, Project 74242-CSM Report, PNNL, February 2021.

[2] L. R. Greenwood and C. D. Johnson, *Least-Squares Neuron Spectral Adjustment with STAYSL PNNL*, International Symposium on Reactor Dosimetry 15, EPJ Web of Conferences Vol 106, 586-594, Aix-en-Provence, France, May 2015, ISBN:978-1-5108-1940-5.

[3] A. Trkov, P.J. Griffin, S.P. Simakov, L.R. Greenwood, et al, *IRDFF-II: A New Neutron Metrology Library*, Nuclear Data Sheets, 163, pp 1-108, 2020.

[4] L. R. Greenwood and R. K. Smither, *SPECTER: Neutron Damage Calculations for Materials Irradiations*, ANL/FPP-TM-197, January 1985.