DISCLAIMER

ORNL/SPR-2025/4204
M3LW-25OR0402013

Light Water Reactor Sustainability Program

CHARPY IMPACT CHARACTERIZATION OF SURVEILLANCE SPECIMENS HARVESTED FROM PALISADES HIGH FLUENCE A-60 CAPSULE

Mikhail A. Sokolov
Materials Science and Technology Division
Oak Ridge National Laboratory

September 2025

U.S. Department of Energy
Office of Nuclear Energy

ORNL/SPR-2025/4204

LIGHT WATER REACTOR SUSTAINABILITY PROGRAM
MATERIALS RESEARCH PATHWAY

CHARPY IMPACT CHARACTERIZATION OF SURVEILLANCE SPECIMENS HARVESTED FROM PALISADES HIGH FLUENCE A-60 CAPSULE

Mikhail A. Sokolov

Materials Science and Technology Division
Oak Ridge National Laboratory

Milestone Report: M3LW-25OR0402013

September 2025

Prepared by
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, TN 37831
managed by
UT-BATTELLE LLC
for the
US DEPARTMENT OF ENERGY
under contract DE-AC05-00OR22725

CONTENTS

CONTENTS.....4

EXECUTIVE SUMMARY.....7

ACKNOWLEDGEMENT.....8

1. INTRODUCTION.....9

2. CHARPY TESTING RESULTS.....12
   - 2.1 Standard Reference Material.....12
   - 2.2 Base Metal.....13
   - 2.3 Weld metal.....14

3. SUMMARY AND DISCUSSION.....16
   - 3.1 Next steps.....17

4. REFERENCES.....18

List of Figures

Figure 1. Arrangement of surveillance capsules in the Palisades reactor vessel [2]. 10

Figure 2. Charpy impact test data of SRM from A-60 capsule in comparison with unirradiated data and previously reported W-110 capsule results. 13

Figure 3. Charpy data of A302B Modified surveillance base metal in PNGS. 14

Figure 4. Charpy data of Palisades beltline surveillance weld. 15

Figure 5. Relationship between radiation embrittlement (ΔT_{41J}) and hardening ($\Delta \sigma_Y$). 17

List of Tables

Table 1. Summary of primary radiation-sensitive elements for the PNGS surveillance materials. 11

Table 2. Charpy impact test results of SRM. 12

Table 3. Charpy impact data of A302B-B Modified specimens from capsule A-60. 13

Table 4. Charpy impact data of Linde 1092 surveillance weld irradiated in capsule A-60. 15

Table 5. Tensile properties of surveillance specimens harvested from PNGS A-60 capsule [6]. 16

EXECUTIVE SUMMARY

Located on the shores of Lake Michigan, the Palisades Nuclear Generating Station (PNGS) was a nuclear power plant that operated in Covert Township, Michigan. The plant had a single pressurized water reactor that produced electricity for the region. The PNGS was shut down in 2022 after more than four decades of service. The PNGS included in its surveillance program a surveillance capsule, designated A-60, containing specimens of a weld metal with nickel content of about 1.36 wt% and copper content of about 0.25 wt%. The capsule was removed from its surveillance position in early 1995 and has been resident in the spent fuel pool since that time. This capsule was irradiated to a fluence of 1.96×10^{20} n/cm^2 (E> 1MeV) that is equivalent for more than 150 effective full power years (EFPYs) for the US reactor pressure vessel (RPV) fleet. The material is also of special interest because of its very high nickel content and potential for development of NiMnSi (nickel-manganese-silicon) precipitates. Combination of very high fluence and very high Ni and Cu content makes the material in this capsule of great interest as benchmark for currently developing embrittlement trend curves (ETC) aiming to predict embrittlement at high fluences. Multi-year efforts by the Light Water Reactor Sustainability (LWRS) Program personnel from the Materials Research Pathway (MRP) to harvest this capsule finally succeeded in 2023. As a result, the Westinghouse Electric Company (WEC) through contract with Oak Ridge National Laboratory (ORNL) came to PNGS site, retrieved the A-60 capsule, brought it to WEC Churchill hot cell facility, opened the capsule and sent all surveillance specimens in the capsule to ORNL for future characterization by July 2023. Total of 9 tensile and 48 Charpy specimens were inventoried in the ORNL hot cells. Testing plan has been developed based on available specimens. It includes hardness, tensile, Charpy impact, Mini-CT fracture toughness testing, in-situ thermal annealing, and microstructural characterization, including Atom Probe Tomography (APT). Charpy impact testing has been completed and results are presented in this report. Moreover, negotiations with Pressurized Water Reactors Owners Group (PWROG) and Westinghouse resulted in Westinghouse donating to ORNL a piece of archive weld and base metal such that unirradiated characterization of these materials can be performed as part of this project. Comparison of measured hardening and embrittlement has been performed and data are compared to available large database.

ACKNOWLEDGEMENT

This research was sponsored by the U.S. Department of Energy, Office of Nuclear Energy, for the Light Water Reactor Sustainability (LWRS) Program Materials Research Pathway. The author extends his appreciation to Timothy Lach and Xiang (Frank) Chen at Oak Ridge National Laboratory (ORNL) for their technical review of this report.

The author is also grateful to Stewart Marsh and Anthony Guajardo, from Materials Science and Technology Division of ORNL for accomplishing this challenging task.

1. INTRODUCTION

The Palisades Nuclear Generating Station (PNGS) was a nuclear power plant located on Lake Michigan, in Van Buren County's Covert Township, Michigan. Built between 1967 and 1970, Palisades was approved to operate at full power in 1973 [1]. The plant's original license was due to expire on March 24, 2011. An application for 20-year extension was filed in 2005 with the U.S. Nuclear Regulatory Commission. It was granted on January 18, 2007. Therefore, the plant was then scheduled for decommissioning by 2031. However, Entergy, the previous operating company, closed the Palisades plant in May 2022 and sold it to Holtec International in June 2022.

The PNGS included in its surveillance program a surveillance capsule, designated A-60. The capsule was removed from its surveillance position in early 1995 and has been resident in the spent fuel pool since that time. This capsule was irradiated to a fluence of 1.96×10^{20} n/cm^2 (E> 1MeV) that is equivalent for more than 150 effective full power years (EFPYs) for the US reactor pressure vessel (RPV) fleet. Combustion Engineering, Inc., the PNGS designer, designed and furnished the RPV surveillance program [2]. It was designed in accordance with ASTM Standard E185-66, "Recommended Practice for Surveillance Tests on Structural Materials in Nuclear Reactors" [3]. The original program included ten capsules to monitor the effects of both neutron and thermal environments on the beltline materials of the RPV. Six capsules were located at the beltline near the inside surface of the RPV, while two capsules intended for accelerated neutron exposure were located on the outer wall of the core support barrel, thus, closer to the core. The remaining two capsules were in a very low flux region above the core for monitoring the effects of thermal exposure (thus, designated as thermal aging capsules). Figure 1 shows a schematic diagram of the capsule locations. All ten capsules contained Charpy V-Notch (CVN) impact and tensile specimens.

One of the ten original capsules, including A-60, contain specimens made from 1) standard reference material (SRM) (also designated a correlation monitor material) from Heavy-Section Steel Technology (HSST) A533-B Plate 01, 2) intermediate shell course A302-B Modified plate D-3803-1, 3) heat-affected-zone (HAZ) material fabricated by welding intermediate shell plates D-3803-2 and D-3803-3 with a submerged arc process using Linde 1092 flux, and 4) weld metal fabricated by welding intermediate shell plates D-3803-1 and D-3803-2 with a submerged arc process using Linde 1092 flux and with both a MIL-B4 electrode and a 1/16-inch diameter Nickel-200 wire feed.

Table 1 provides a summary of the primary chemical elements responsible for radiation sensitivity in the four materials available in the surveillance capsules. This Table illustrates somewhat interesting combination of major chemical elements responsible for radiation embrittlement, namely copper and nickel, in these surveillance materials. Surveillance weld has very high nickel and copper contents. Base metal, A302B Mod. plate, has high copper and typical nickel content. The SRM metal, A533B plate, has intermittent copper and typical nickel content. In other words, these surveillance materials will cover important combinations of copper/nickel contents that are of interest to provide benchmark data for tailoring existing and potential new embrittlement trend correlation (ETC) models given extremely high fluence range for surveillance specimens. In addition, chemical composition of three beltline welds in Palisades RPV are also provided in this Table for comparison. The more detailed description of this capsule content and harvesting efforts to bring these specimens to the ORNL are provided in the previous LWRS milestone reports [4,5].

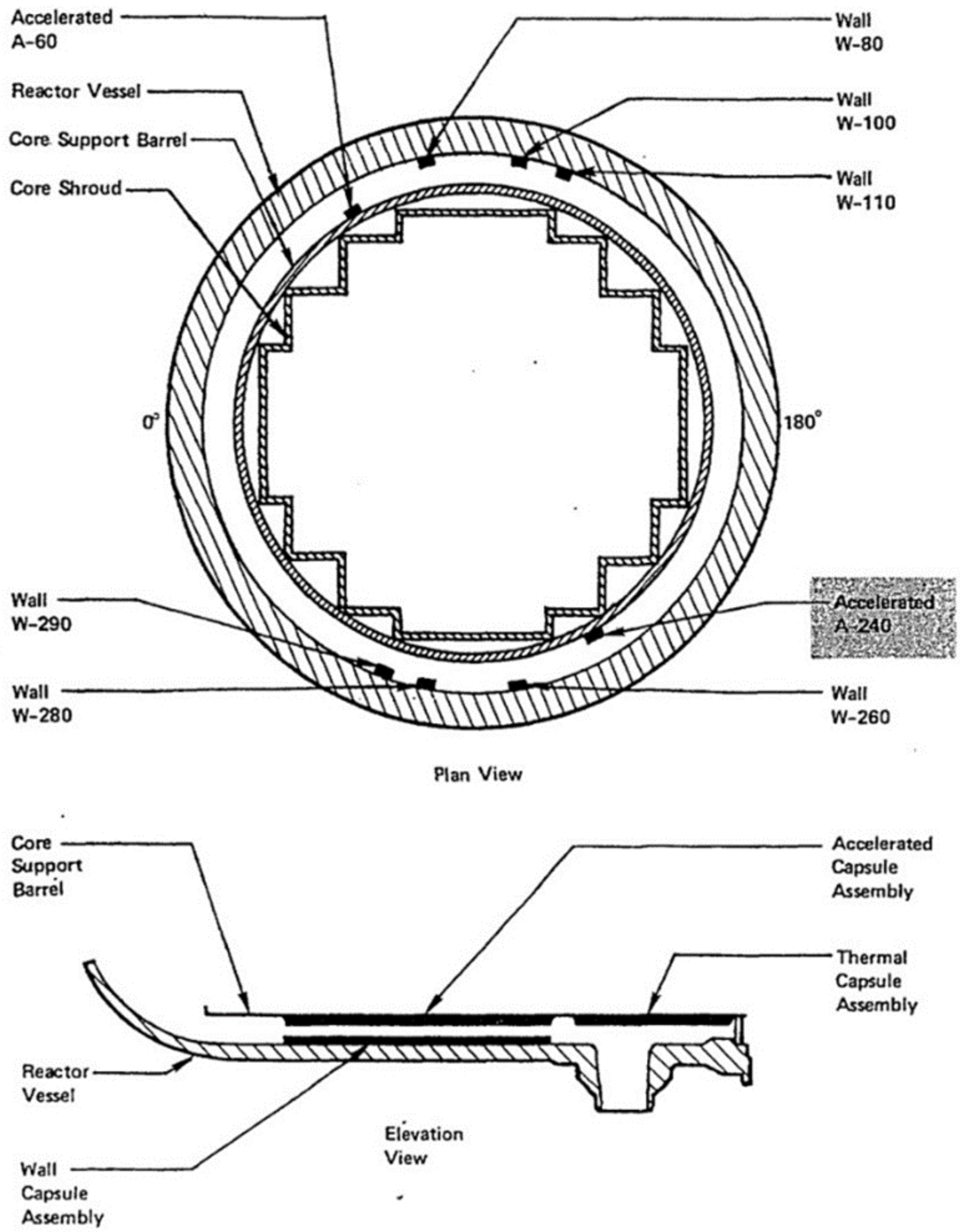

Figure 1. Arrangement of surveillance capsules in the Palisades reactor vessel [2].

Tensile specimens have been tested, analyzed, and previously reported [6]. In the current report, results of Charpy impact testing are presented and compared to tensile data from the previous report to satisfy LWRS Milestone report M3LW-25OR0402013, entitled "Complete tensile and Charpy impact characterization of surveillance specimens harvested from Palisades high fluence A-60 capsule."

Table 1. Summary of primary radiation-sensitive elements for the PNGS surveillance materials.

Material	Heat Designation	Chemical Composition, wt%				Chemistry Factor US NRC Reg. Guide 1.99, Rev. 2, °F
		Copper	Nickel	Manganese	Silicon	
Base Metal, Plate A302B, Mod.	D-3803-1	0.23	0.51	1.37	0.18	156
Surveillance Weld, Linde 1092	3277	0.25	1.36	1.23	0.20	272*
Weld Heat-Affected-Zone	3277	0.25	1.10	1.23	0.20	258
Intermediate and Lower Shell Longitudinal Weld, Linde 1092	W5214	0.307	1.045	1.161	0.252	260
Lower Shell Longitudinal Weld, Linde 1092	34B009	0.185	1.121	1.269	0.181	231
Circumferential Weld, Linde 124	27204	0.194	1.067	1.281	0.217	226
Standard Reference, A533B-1 Plate	HSST Plate 01	0.17	0.66	1.46	0.19	129

* The chemistry factor is limited by the maximum nickel content of 1.20 wt % in US NRC Reg. Guide 1.99 Rev. 2 [7].

2. CHARPY TESTING RESULTS

Four groups of Charpy specimen were included in A-60 capsule. Standard Reference Material (12 specimens), weld metal (12 specimens), base metal (12 specimens), and heat-affected zone HAZ material (12 specimens). It was decided not to test HAZ specimens and save them for future consideration since those specimens do not represent direct interest for the use in benchmarking ETC models.

2.1 Standard Reference Material

The HSST A-533B Plate 01 was used as the standard reference material within PNGS surveillance program. The Charpy test data from SRM specimens in capsule A-60 are presented in Table 2.

Table 2. Charpy impact test results of SRM.

Specimen ID	Test Temp.		Dial Energy		Lateral Expansion		Fracture Appearance
	°F	°C	ft-lbs	J	mils	mm	% Shear
51A	72	22	2.75	3.7	0	0.00	0
51B	212	100	7.20	9.8	7	0.18	10
51C	392	200	36.10	48.9	26	0.66	55
51L	527	275	73.03	99.0	46	1.17	100
51M	392	200	43.43	58.9	27	0.69	60
51P	302	150	18.33	24.9	14	0.36	35
51T	302	150	13.29	18.0	10	0.25	35
51U	347	175	23.57	32.0	21	0.53	40
51Y	347	175	28.61	38.8	16	0.41	45
515	347	175	25.79	35.0	22	0.56	40
516	392	200	66.44	90.1	33	0.84	70
517	545	285	79.08	107.2	47	1.19	100

After irradiation at such high fluence, the SRM material exhibited shift of Charpy T_{41J} transition temperature of 188°C. The upper-shelf energy (USE) dropped by 77 Joules but still remained above 100J level. The Charpy data from A-60 capsule and unirradiated data are presented in Figure 2. The Charpy specimens of SRM material were also included in previously tested and reported [8] W-110 capsule and included in Figure 2 for comparison purposes. Shift of T_{41J} transition temperature in A-60 capsule (188°C) is more the twice higher than after irradiation in W-110 capsule (86°C) to $1.64x10^{19}$ n/cm^2.

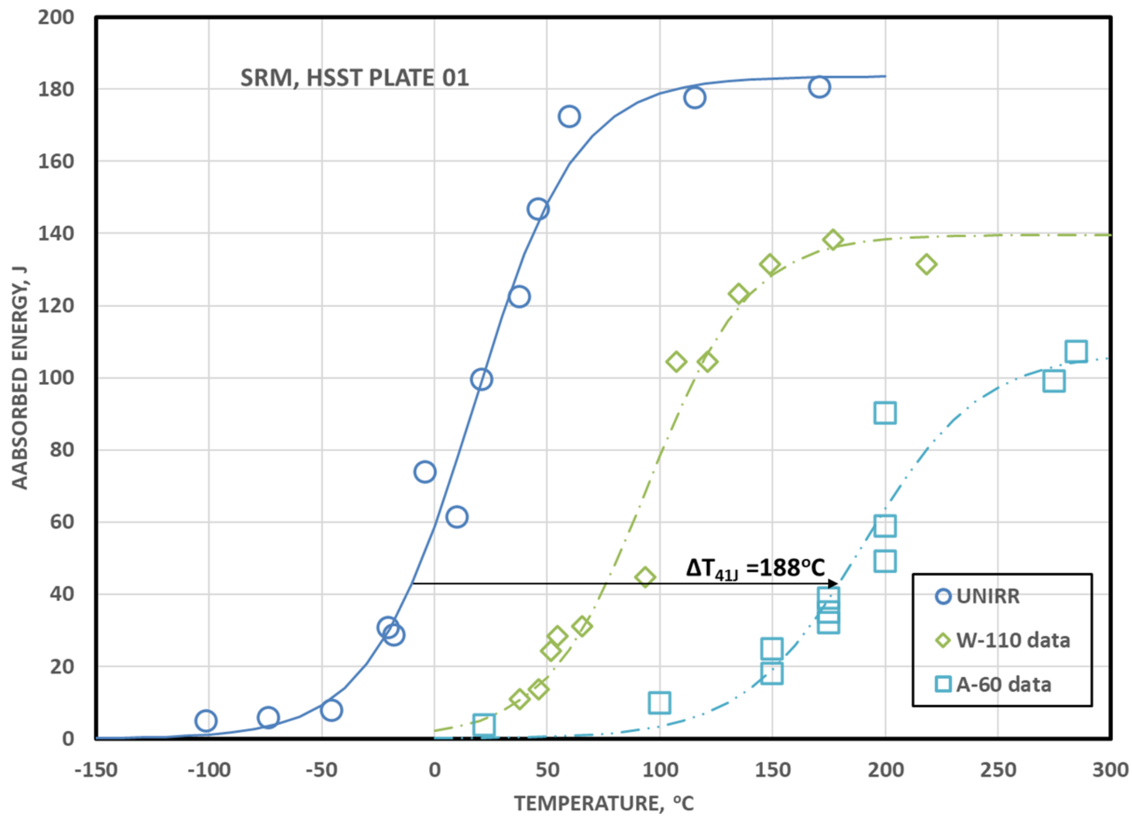

Figure 2. Charpy impact test data of SRM from A-60 capsule in comparison with unirradiated data and previously reported W-110 capsule results.

2.2 Base Metal

Charpy impact test results of A302-B Modified plate D-3803-1 irradiated in capsule A-60 are presented in Table 3.

Table 3. Charpy impact data of A302B-B Modified specimens from capsule A-60.

Specimen Id.	Test Temp.		Dial Energy		Lateral Expansion		Fracture Appearance
	°F	°C	ft-lbs	J	mils	mm	% Shear
111	392	200	82.77	112.2	42	1.07	100
112	212	100	7.32	9.9	7	0.18	20
113	302	150	30.46	41.3	24	0.61	45
114	347	175	67.76	91.9	44	1.12	70
115	302	150	38.04	51.6	30	0.76	55
116	73	23	2.62	3.6	10	0.25	0

11D	212	100	4.78	6.5	8	0.20	10
11E	482	250	87.23	118.3	65	1.65	100
11J	257	125	12.28	16.6	10	0.25	25
11K	302	150	31.91	43.3	27	0.69	50
11L	257	125	12.97	17.6	12	0.30	20
11M	257	125	14.56	19.7	15	0.38	N/A

Figure 3 summarizes the Charpy base metal data from A-60 capsule as well as previously reported unirradiated data [9] and data from previous capsules [8, 10-12]. The Charpy T_{41J} shift was 165°C. The USE dropped by 95J, and yet remained at relatively high level of 121J. Four previously tested capsules [8,10-12] were exposed to fluences ranging from 0.94×10^{19} to 4.09×10^{19} n/cm^2 (E>1Mev). Noticeably, A-60 capsule base metal data still exhibited a relatively high degree of radiation embrittlement suggesting lack of saturation.

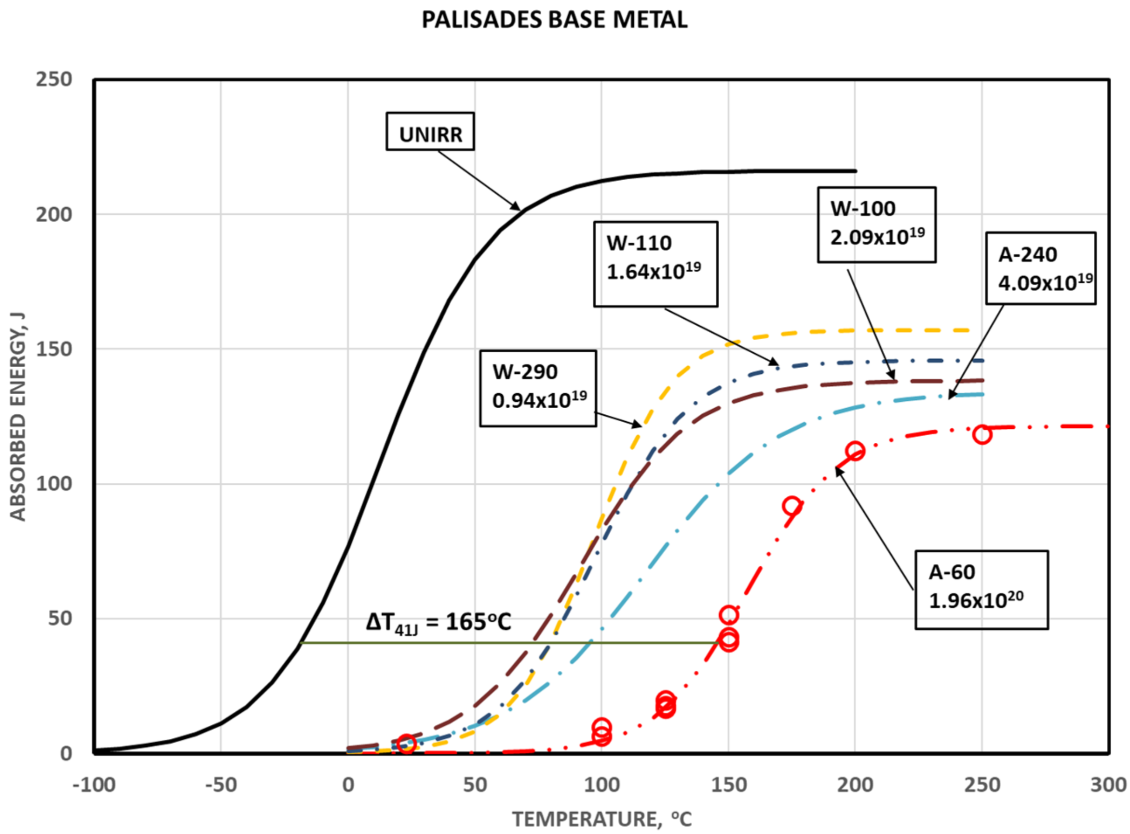

Figure 3. Charpy data of A302B Modified surveillance base metal in PNGS.

2.3 Weld metal

Linde 1092 was used as the surveillance beltline weld within original PNGS surveillance program. The Charpy test data from Linde 1092 weld specimens in capsule A-60 are presented in Table 4. Comparison of A-60 and unirradiated [9] and previously tested other surveillance capsules [8, 10-12] data are illustrated in Figure 4. The Charpy T_{41J} shift was 243°C after irradiation in A-60 capsule. The USE dropped by 93J to 67J, just missing the 68J (50 ft-lb) 10CRF50 mark. Four previously tested capsules [8,10-12] were exposed to fluences ranging from 0.94×10^{19} to 4.09×10^{19} n/cm^2 (E>1Mev).

Table 4. Charpy impact data of Linde 1092 surveillance weld irradiated in capsule A-60.

Specimen Id.	Test Temp.		Dial Energy		Lateral Expansion		Fracture Appearance
	°F	°C	ft-lbs	J	mils	mm	% Shear
311	545	285	49.33	66.9	39	0.99	100
312	302	150	21.49	29.1	13	0.33	60
313	392	200	33.11	44.9	29	0.74	75
314	347	175	31.85	43.2	25	0.64	70
315	392	200	46.68	63.3	36	0.91	90
316	302	150	22.60	30.6	17	0.43	55
317	347	175	34.10	46.2	27	0.69	75
31A	347	175	24.16	32.8	27	0.69	60
31B	122	50	7.13	9.7	4	0.10	5
31M	392	200	31.65	42.9	25	0.64	80
31P	545	285	46.40	62.9	32	0.81	100
31T	302	150	11.20	15.2	11	0.28	30

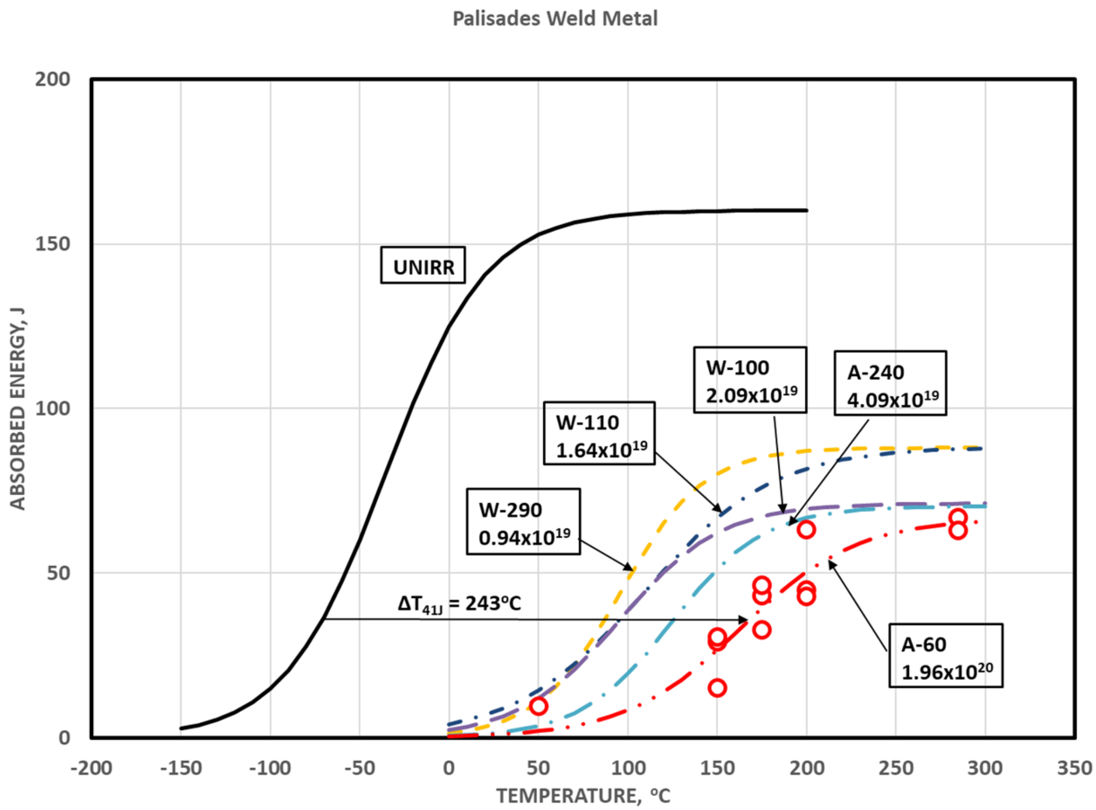

Figure 4. Charpy data of Palisades beltline surveillance weld.

3. SUMMARY AND DISCUSSION

As expected, the Charpy data from PNGS high fluence surveillance A-60 capsule confirmed very high level of embrittlement of materials included in this capsule. The beltline weld exhibited uniquely high shift of the Charpy transition temperature, 243°C. One of the first objectives of this study is to verify that these high fluence data are in general agreement with previous surveillance data and correlations between embrittlement and hardening. Accomplishment of this objective would justify using A-60 data as a benchmark for various currently accepted and newly developed embrittlement trend correlation (ETC) to rationalize use of such ETC for long-term operation extrapolations.

Table 5. Tensile properties of surveillance specimens harvested from PNGS A-60 capsule [6].

Material	Specimen ID	STRENGTH		Elongation		Reduction of Area, %
		Yield, MPa	Ultimate, MPa	Uniform, %	Total, %	
Base Metal	1D2	772	866	5.8	12.9	51.3
	1D6	769	860	5.6	13.3	54.5
Weld Metal	3D4	966	1005	4.7	10.6	43.4
	3D5	929	974	4.8	10.2	37.9
HAZ	4D1	807	895	2.8	9.2	53.3

Very high level of embrittlement of surveillance Charpy specimens in A-60 capsule is in general agreement with previously reported tensile data from this capsule, see Table 5 [6]. Based on Table 5 and unirradiated data [9], radiation hardening ($\Delta\sigma_Y$) was 330 MPa and (an enormous) 504 MPa for base and weld metals, respectfully. This yields the ratio of embrittlement to hardening ($\Delta T_{41J}/\Delta\sigma_Y$) equal to 0.50 and 0.48 for A-60 surveillance capsule base and weld metals, respectfully. It is a common practice to compare these results with ratio of radiation embrittlement to radiation hardening from [13]:

$$\Delta \boldsymbol{T_0} = \mathbf{0.7}\Delta\boldsymbol{\sigma_Y} \quad (1)$$

where ΔT_0 is the shift of the reference fracture toughness transition temperature. The A-60 data for both base and weld metals are somewhat lower than suggested by Eq. (1). It needs to point out that Eq. (1) was established with shifts of fracture toughness reference temperature, while Charpy 41J transition temperatures were measured in this phase of A-60 characterization. Reference 13 did not specifically address the $\Delta T_{41J}/\Delta\sigma_Y$ ratio, even though it provided a correlation between ΔT_0 and ΔT_{41J} which is not always 1:1. Thus, the data from [13] have been revisited in this report such that $\Delta T_{41J}/\Delta\sigma_Y$ could be established. Moreover, $\Delta T_{41J}/\Delta\sigma_Y$ data from the power reactor embrittlement database (PR-EDB) [14] have been included in the current analysis to improve and increase overall database. The resulting expression yielded the following ratio:

$$\Delta \boldsymbol{T_{41J}} = \mathbf{0}.58\Delta\boldsymbol{\sigma_Y} \quad (2)$$

Results of this analysis are presented in Figure 5. Data from Palisades surveillance capsules are included as well in red color. This plot clearly illustrates how much Palisades A-60 data are outside of the available RPV database to emphasize the importance of these high fluence data to verify extrapolation of current correlations to long term operation conditions. Based on Figure 5, the Palisades surveillance base and weld metal data are within the scatter band of the available database.

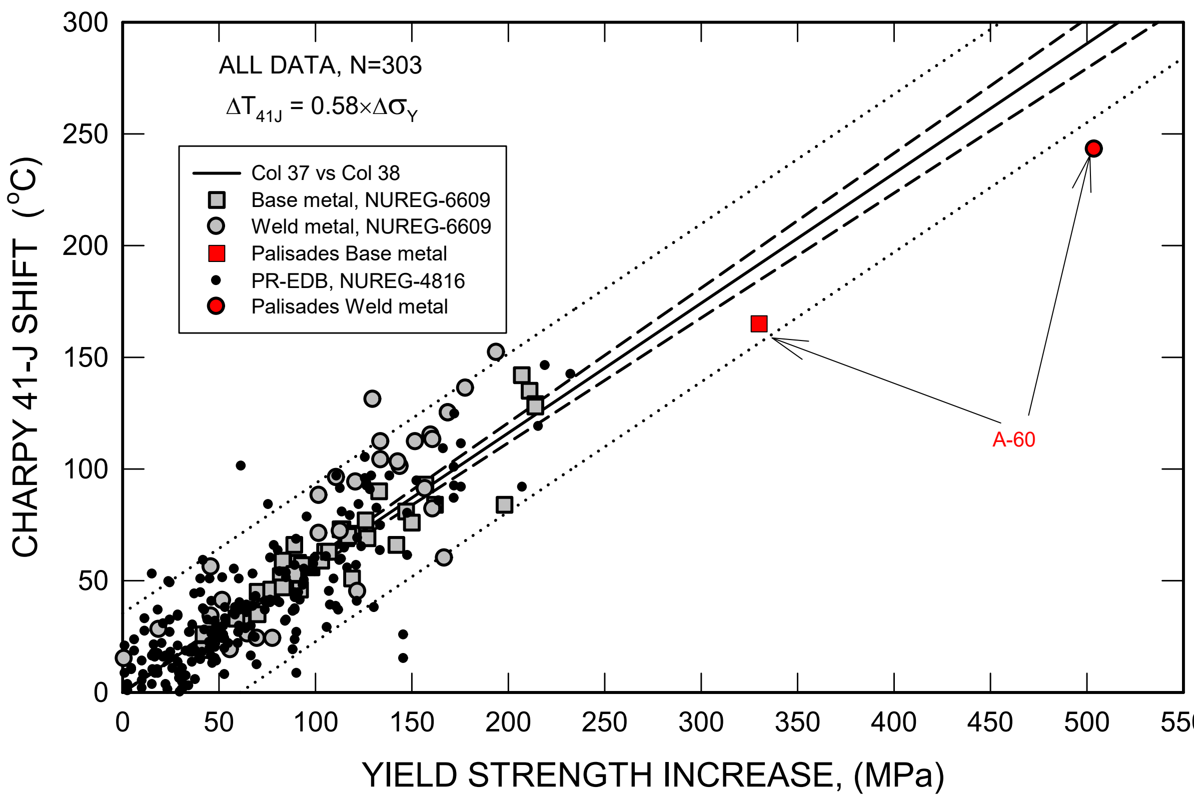

Figure 5. Relationship between radiation embrittlement (ΔT_{41J}) and hardening ($\Delta\sigma_Y$).

The measured Charpy shifts allowed to evaluate A-60 data relatively pressurized thermal shock (PTS) screening criteria in 10CFR50.61. According to 10CFR50.61(b)(2), RT_{NDT} of the RPV base metal must not exceed RT_{PTS} 270°F (132°C). The measured $RT_{NDT(U)}$ was reported [9] as -10°F. Shift of Charpy 41J transition temperature is the measure of RT_{NDT} shift as result of irradiation. Given the Charpy shift of the base metal in Section 2.2, the irradiated RT_{NDT} of the base metal is 287°F, exceeding RT_{PTS} criteria by only 17°F despite very high fluence. The RT_{NDT} of the surveillance weld was not determined in [9] while Charpy transition temperature at 50 ft-lb (68J) was reported as -50°F. In the current analysis, it was assumed that RT_{NDT} of the surveillance weld is T_{50ftlb} – 60°F = -110°F in the unirradiated condition. The shift of Charpy T_{41J} is 437°F (243°C, see section 2.3). Thus, RT_{NDT} of Palisades surveillance weld after irradiation in A-60 capsule is 327°F. According to 10CFR50,61(b)(2), RT_{NDT} of the RPV must not exceed RT_{PTS} 270°F (132°C) for longitudinal weld metal and 300°F (149°C) for circumferential weld. Palisades surveillance weld in A-60 capsule exceeded both PTS criteria. The PTS screening criteria exercise above suggests that for nuclear power plants with RPV that contain similar radiation sensitive materials, some mitigation techniques, like thermal annealing for example, might be necessary in case of interest in very long-term operation. Moreover, the RT_{NDT} exercise for A-60 irradiated base and weld metals was performed for illustration purposes rather than following every step in 10CRF50.61(b) without adding required margins, using generic value of RT_{NDT}, etc.

3.1 Next steps

To complete the characterization and analysis of the A-60 capsule data, several steps need to be performed. One of important steps is to machine Mini-CT specimens and perform fracture toughness evaluation of A-60 materials. Comparison of measured shifts of the transition temperatures will also be performed to confirm relationship between fracture toughness and Charpy shifts at very high fluences. It was discovered during this phase of evaluation that surveillance weld has very wide range of nickel variation based on several measurements made in different locations and at different times. Decision was made to use available archived surveillance weld to perform chemical analysis in locations where actual surveillance specimens were made. That would allow us to use real chemical composition values to compare predictions of various ETC with measured shifts of Charpy of fracture toughness.

4. REFERENCES

[1]. Wikipedia contributors, "Palisades Nuclear Generating Station," *Wikipedia, The Free Encyclopedia,* https://en.wikipedia.org/wiki/Palisades_Nuclear_Generating_Station (accessed August 16, 2023).

[2]. R. C. Groeschel, *"Summary Report on Manufacture of Test Specimens and Assembly of Capsules for Irradiation Surveillance of Palisades Reactor Vessel Materials, " CE Report No. P-NLM-019*, Combustion Engineering, Inc., Windsor, Connecticut, April 1, 1971.

[3]. ASTM Standard E 185-66, *"Recommended Practice for Surveillance Tests on Structural Materials in Nuclear Reactors,"* American Society for Testing and Materials, Philadelphia, Pennsylvania.

[4]. R.K. Nanstad, M.A. Sokolov, and W.L. Server, "*Plan for Evaluation of Reactor Pressure Vessel Surveillance Materials from Palisades Nuclear Generating Station*," ORNL/TM-2020/1444, Oak Ridge National Laboratory, Oak Ridge, February 2020.

[5]. M.A. Sokolov, X. Chen, T. Rosseel, B. Hall, and G. Wiggins, "*Report on Retrieval of the Reactor Pressure Vessel Surveillance Capsule A-60 from Palisades Nuclear Generating Station,*" ORNL/ LTR-2023/3012, Oak Ridge National Laboratory, Oak Ridge, August 2023.

[6]. M.A. Sokolov and X. Chen, "Initial Tensile Test Results of Surveillance Specimens Harvested from High-Fluence A-60 Capsule from Palisades Nuclear Generating Station," ORNL/SPR-2024/3545, Oak Ridge National Laboratories, Oak Ridge, August 2024.
[7]. US Nuclear Regulatory Commission. Regulatory Guide 1.99, Revision 2, May 1988.
[8]. Analysis of capsule W-110 from the Consumers Power Company Palisades Reactor Vessel Radiation Surveillance Program, WCAP-14014, May 1994.
[9]. J.S. Perrin and E.O. Fromm, "Palisades Pressure Vessel Irradiation Capsule Program: Unirradiated Mechanical Properties," Battelle, August 25, 1977.
[10]. M.K. Kunka and CA. Cheney, "Analysis of capsules T-330 and W-290 from the Consumers Power Company Palisades Reactor Vessel Radiation Surveillance Program," WCAP-10637, September 1984.
[11]. Analysis of capsule W-100 from the Nuclear Management Company Palisades Reactor Vessel Material Surveillance Program, BWXT, February 2004.
[12]. J. S. Perrin et. al., "Palisades Nuclear Plant Reactor Pressure Vessel Surveillance Program: Capsule A-240," BCL-585-12, Battelle Columbus Laboratories, Columbus, Ohio 43201, March 13, 1979.
[13]. M.A. Sokolov and R.K. Nanstad, "Comparison on Irradiation-Induced Shifts of K_{Jc} and Charpy Impact Toughness for Reactor Pressure Vessel Steels," pp. 167-190 in Effects of Radiation on Materials: 18th International Symposium, ASTM STP 1325, R. K. Nanstad, M. L. Hamilton, F. A. Garner, and A. S. Kumar, Eds., American Society for Testing and Materials, West Conshohocken, Pa., 1999.
[14]. F. W. Stallmann, J. A. Wang, F. B. K. Kam, and B. J. Taylor, "PR-EDB: Power Reactor Embrittlement DataBase, Version 2," NUREG/CR-4816 (1994).