

Light Water Reactor Sustainability Program

Initial Tensile Test Results of Surveillance Specimens Harvested from High-Fluence A-60 Capsule from Palisades Nuclear Generating Station

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**LIGHT WATER REACTOR SUSTAINABILITY PROGRAM
MATERIALS RESEARCH PATHWAY**

**INITIAL TENSILE TEST RESULTS OF SURVEILLANCE SPECIMENS HARVESTED
FROM HIGH-FLUENCE A-60 CAPSULE FROM PALISADES NUCLEAR
GENERATING STATION**

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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 the early 1995 and has been resident in the spent fuel pool since that time. This capsule was irradiated to a fluence of 1.87×10^{20} n/cm² (E > 1MeV) that is equivalent for more than 120 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 the 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). Initial tensile 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.

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The authors are also grateful to Guy Wiggins from Holtec International, Stewart Marsh, Anthony Guajardo, and Matt Stanford from 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 licensee 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, containing specimens of a weld metal with nickel content of about 1.36 wt% and copper content of about 0.20 wt%. The capsule was removed from its surveillance position in the early 1995 and has been resident in the spent fuel pool since that time. This capsule was irradiated to a fluence of 1.87×10^{20} n/cm² (E > 1MeV) that is equivalent for more than 120 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.

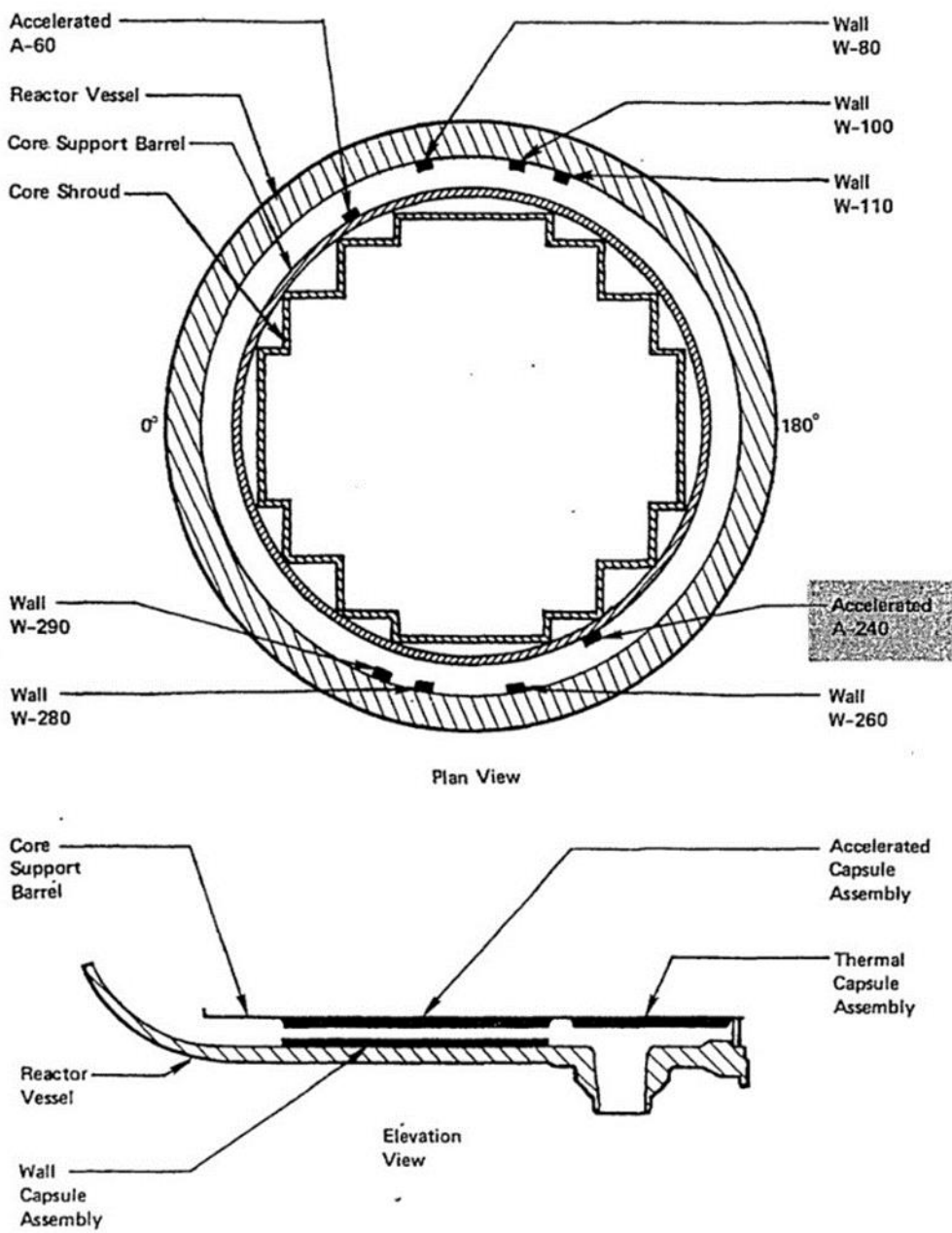


Figure 1. Arrangement of surveillance capsules in the Palisades reactor vessel [2].

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].

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 potential new ETC models for high fluence range.

This report is to satisfy LWRS Milestone report M2LW- 24OR0402013, entitled “Complete inventory and testing plan for specimens harvested from Palisades high fluence surveillance 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
		Copper	Nickel	Manganese	Silicon	
Base Metal, Plate A302B, Mod.	C1279-3	0.23	0.51	1.37	0.18	154
Surveillance Weld, Linde 1092	3277	0.25	1.36	1.23	0.20	214*
Heat-Affected-Zone	3277	0.25	1.10	1.23	0.20	211
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.

2. TENSILE TESTING RESULTS

Total of 9 tensile specimens were included in the high fluence A-60 capsule. Three specimens of each base, weld, and HAZ metals. No SRM tensile were in the capsule. Only two specimens of each base and weld metals were tested up to date. Results were very consistent, and it was decided to save the last specimen from each material for future considerations. Only one HAZ specimen was tested. Results did not reveal any unusual data. Yield and Ultimate Strengths of this HAZ specimen were in between base and weld metals data, leaning closer to the base metal data. Since these HAZ results did not reveal any deviation from overall trend in HAZ surveillance data from other surveillance data, it was decided to save two remaining specimens for future considerations. All tests were performed at room temperature.

As it was mentioned earlier, this capsule was removed from reactor in 1995. Over such long cool down period, the dose rate of these specimens was significantly reduced. As a result, dose rate was lower enough to perform tensile testing of these specimens in the Fracture Mechanics Laboratory at ORNL. Temporary restricted radiological contamination area was set up around a 50-kip MTS servohydraulic frame. Tests were performed with initial loading rate of 0.005 in./min. After passing yield point and/or 0.5% strain, the loading rate was increased to 0.05 in./min. Elongation was measured with calibrated Epsilon displacement gage. Figure 2 illustrate a specimen placed in the grips of the 50-kip MTS frame with Epsilon gage attached inside the anti-contamination containment and ready to be tested.

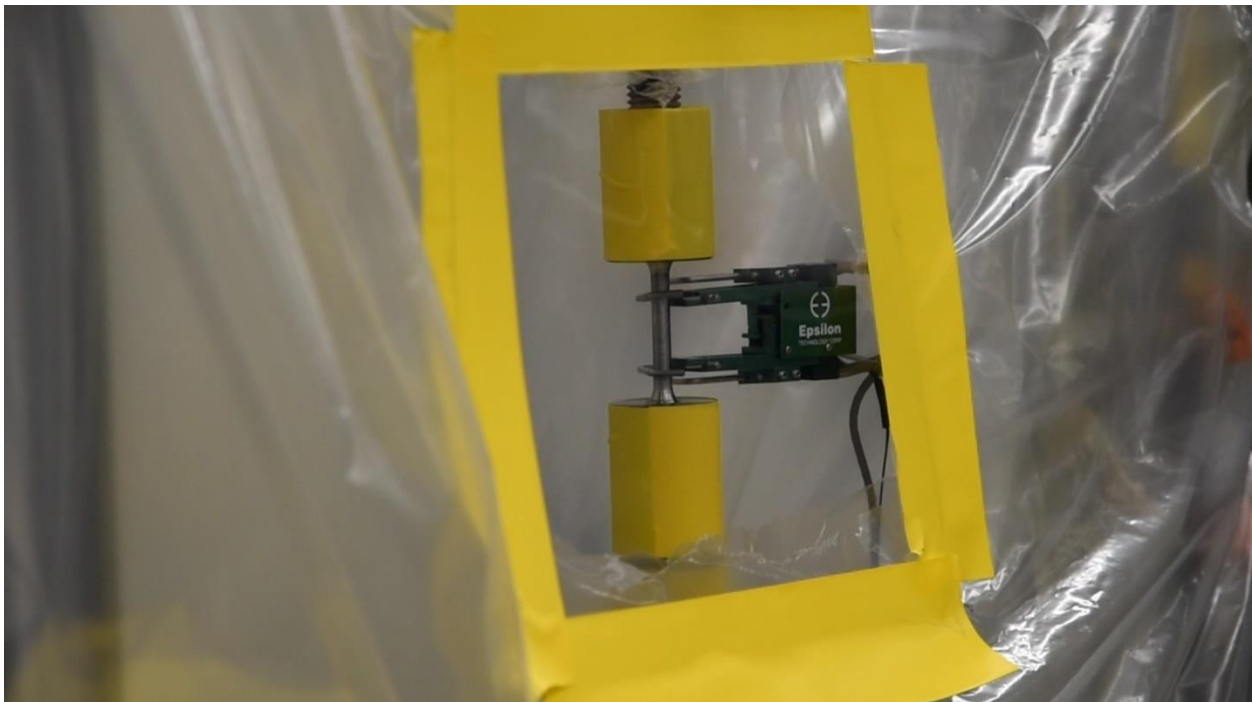


Figure 2. PNGS surveillance specimen 1D6 placed in the grips with Epsilon displacement gage attached.

Figure 3 captured the moment when one technician, Stewart Marsh, was loading a specimen in the grips under supervision of a radiation control technician, Matt Stanford. Both people were inside the radiological area in protective clothes. The second technician, Anthony Guajardo, was controlling frame and data acquisition through computer outside the radiological area. After completion of the tests, the radiological area was removed, and no residual contamination has been left in the Fracture Mechanic Laboratory and its equipment. Table 2 provides a summary of tensile properties of selected surveillance specimens harvested from PNGS A-60 capsule.



Figure 3. A surveillance test specimen from capsule A-60 is loading into MTS test frame inside temporary radiological area.

Table 2. Tensile properties of selected surveillance specimens harvested from PNGS A-60 capsule.

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

Individual stress-strain curves for each specimen tested are represented in Figures 4 through 8.

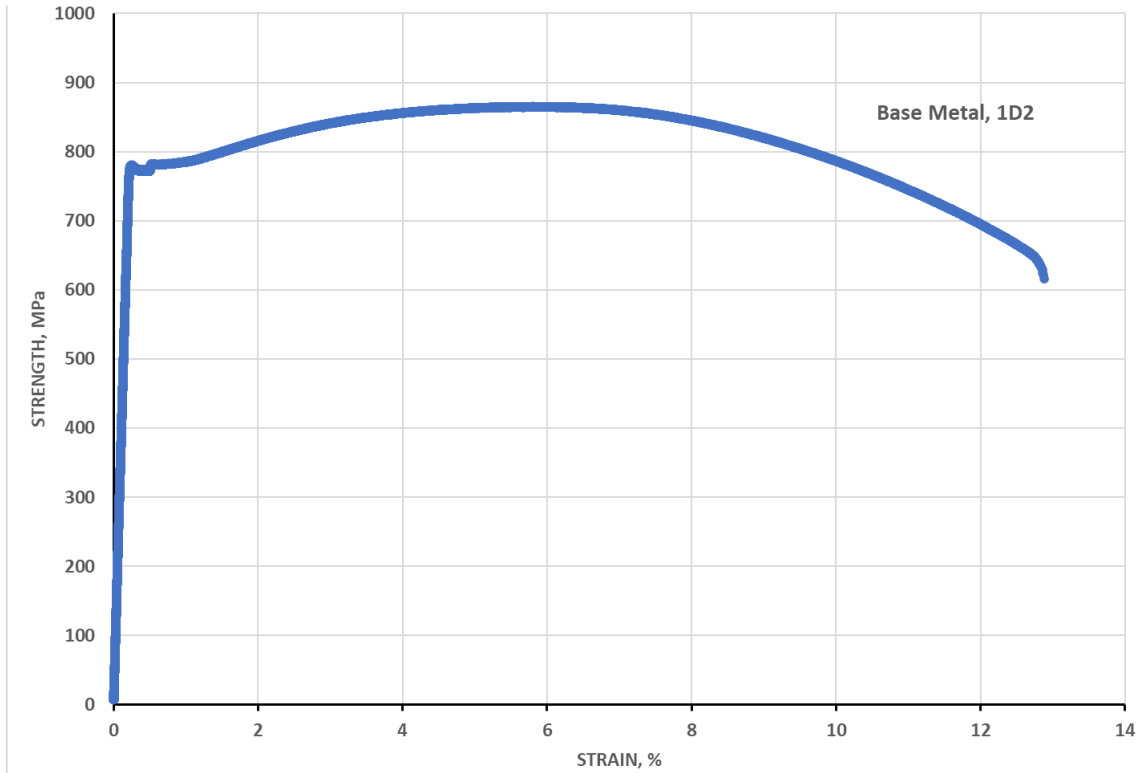


Figure 4. Stress-Strain curve of PNGS surveillance base metal specimen 1D2.

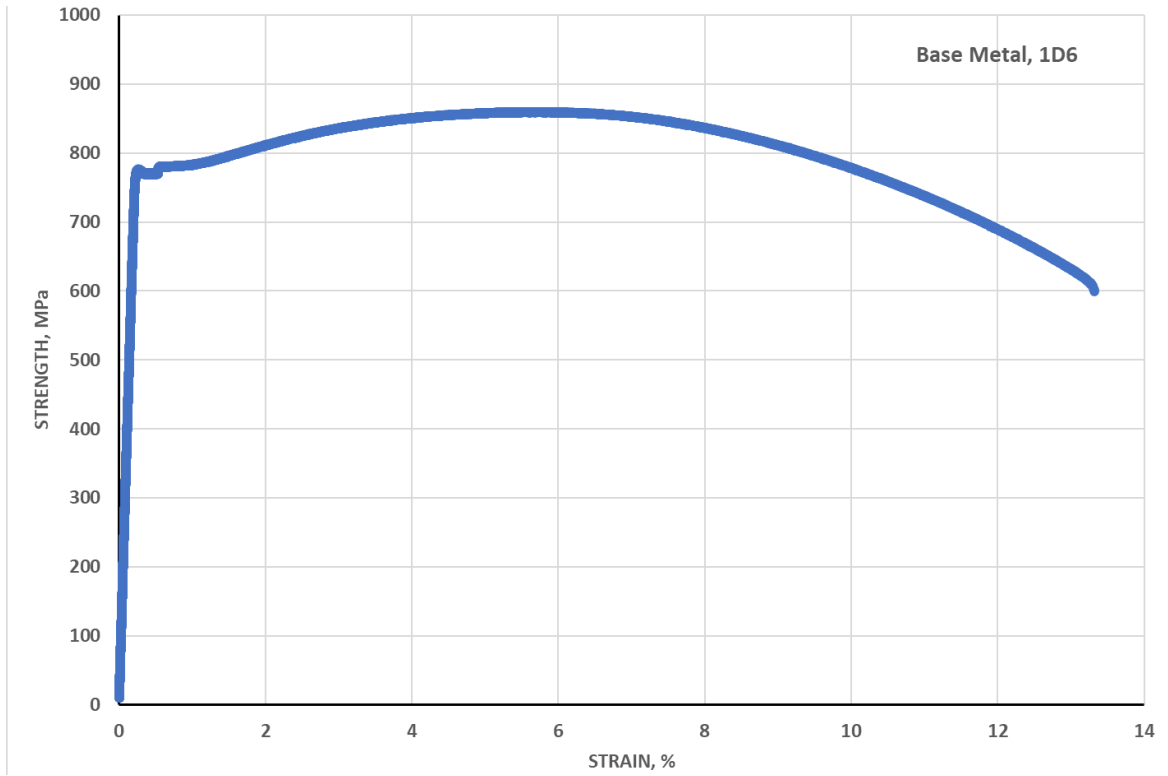


Figure 5, Stress-Strain curve of PNGS surveillance base metal specimen 1D6.

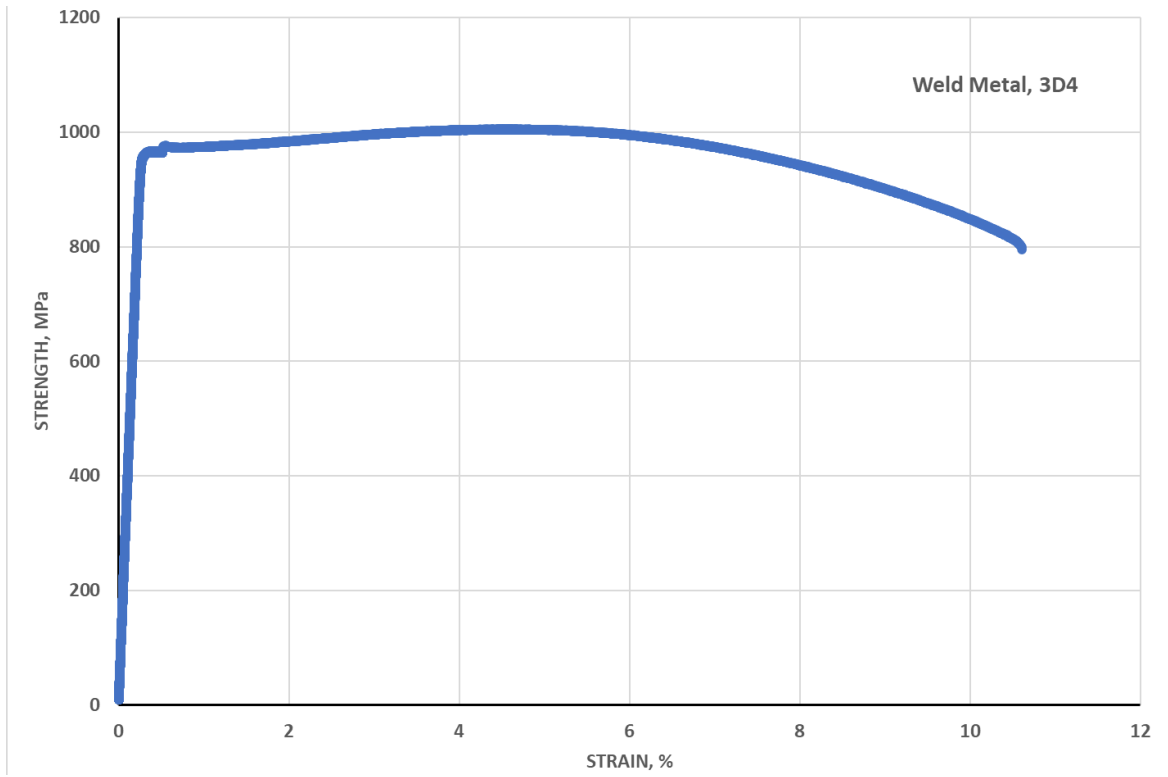


Figure 6. Stress-Strain curve of PNGS surveillance weld specimen 3D4.

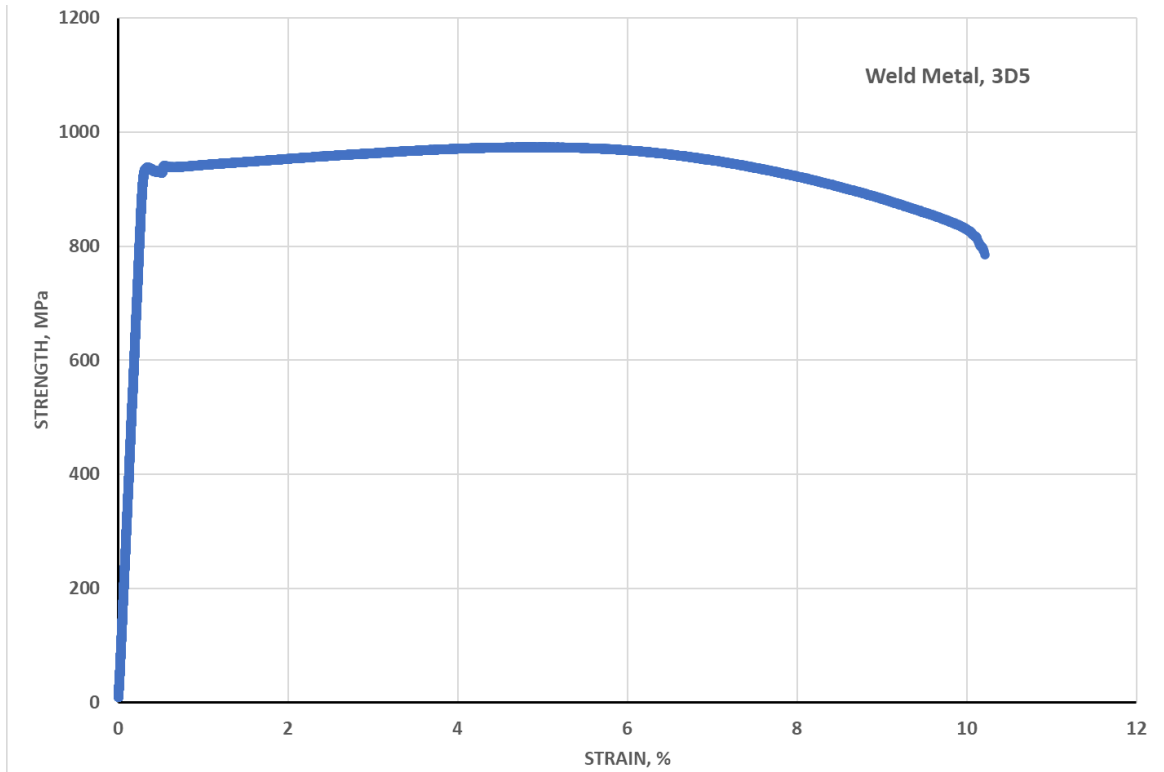


Figure 7. Stress-Strain curve of PNGS surveillance weld metal specimen 3D5.

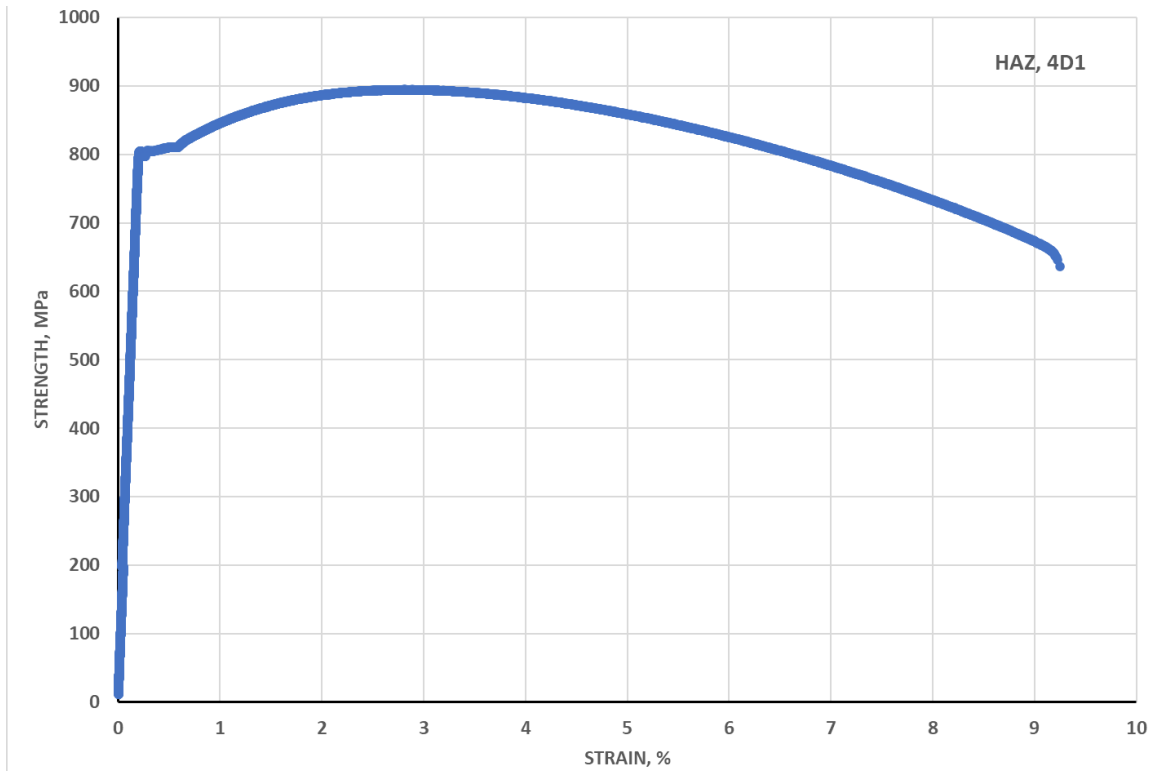


Figure 8. Stress-Strain curve of PNGS surveillance HAZ specimen 4D1.

Both materials, base and weld metals, exhibited very high level of hardening after exposure to such high fluence, $1.87 \times 10^{20} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$). The ultimate strength of weld metal approaches $\sim 1000 \text{ MPa}$ level.

It is anticipated that shift of ductile-to-brittle transition temperature for these materials will be above 200°C and this will help to plan Charpy test temperature selection for A-60 specimens. Figures 9 and 10 compares present data with unirradiated tensile properties of surveillance materials as reported in Ref. 6.

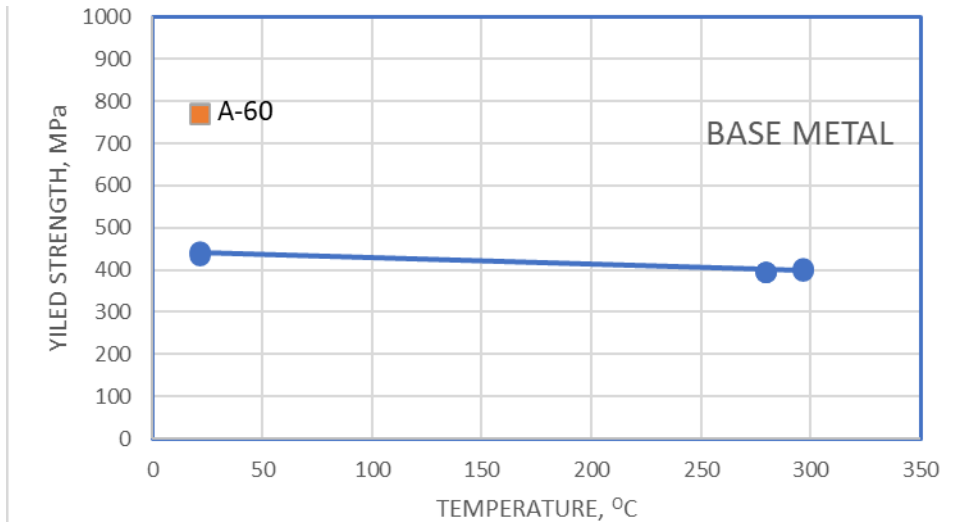


Figure 9. Yield strength of unirradiated [6] and irradiated in A-60 capsule base metal specimens.

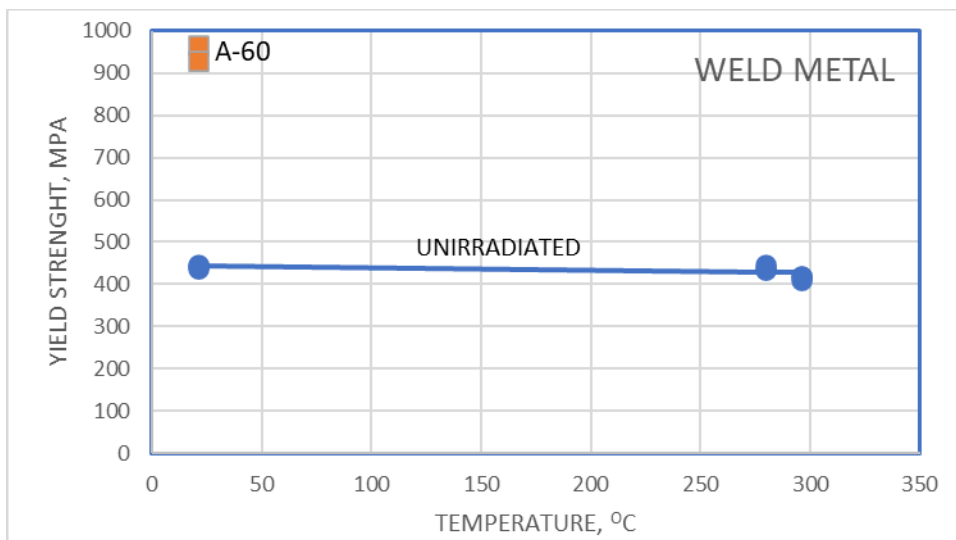


Figure 10. Yield strength of unirradiated [6] and irradiated in A-60 capsule weld metal.

3. SUMMARY

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 the early 1995 and has been resident in the spent fuel pool since that time. This capsule was irradiated to a fluence of 1.87×10^{20} n/cm² (E> 1MeV) that is equivalent for more than 120 EFYs for the US reactor pressure vessel fleet. Specimens from the PNGS high-fluence surveillance capsule A-60 arrived to ORNL in July 2023. They have been inventoried and a testing plan has been developed that includes tensile, Charpy impact, Mini-CT fracture toughness, in-situ thermal annealing, and Atom Probe Tomography characterization. Tensile testing of base, weld, and HAZ specimens has been completed and results are showing that these materials exhibited very high level of hardening after irradiation to 1.87×10^{20} n/cm² (E>1 MeV). These results will be used to select proper test temperatures for Charpy impact testing that will be performed in FY 2025.

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]. J.S. Perrin and E.O. Fromm, "Palisades Pressure Vessel Irradiation Capsule Program: Unirradiated Mechanical Properties," Battelle, August 25, 1977.