ORNL/TM-2015/561

Progress Report on Disassembly and Post-Irradiation Experiments for UCSB ATR-2 Experiment

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ORNL/TM-2015/561

Light Water Reactor Sustainability

PROGRESS REPORT ON DISASSEMBLY AND POST-IRRADIATION EXPERIMENTS FOR UCSB ATR-2 EXPERIMENT

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1. INTRODUCTION

The reactor pressure vessel (RPV) in a light-water reactor (LWR) represents the first line of defense against a release of radiation in case of an accident. Thus, regulations that govern the operation of commercial nuclear power plants require conservative margins of fracture toughness, both during normal operation and under accident scenarios. In the unirradiated condition, the RPV has sufficient fracture toughness such that failure is implausible under any postulated condition, including pressurized thermal shock (PTS) in pressurized water reactors (PWR). In the irradiated condition, however, the fracture toughness of the RPV may be severely degraded, with the degree of toughness loss dependent on the radiation sensitivity of the materials. As stated in previous progress reports, the available embrittlement predictive models, e.g. [1], and our present understanding of radiation damage are not fully quantitative, and do not treat all potentially significant variables and issues, particularly considering extension of operation to 80y.

The major issues regarding irradiation effects are discussed in [2, 3] and have also been discussed in previous progress and milestone reports. As noted previously, of the many significant issues discussed, the issue considered to have the most impact on the current regulatory process is that associated with effects of neutron irradiation on RPV steels at high fluence, for long irradiation times, and as affected by neutron flux. It is clear that embrittlement of RPV steels is a critical issue that may limit LWR plant life extension. The primary objective of the LWRSP RPV task is to develop robust predictions of transition temperature shifts (TTS) at high fluence (ϕt) to at least 10^{20} n/cm^2 (>1 MeV) pertinent to plant operation of some pressurized water reactors (PWR) for 80 full power years. Correlations between the high flux test reactor results and low flux surveillance specimens must be established for proper RPV embrittlement predictions of the current nuclear power fleet. Additionally, a complete understanding of defect evolution for high nickel RPV steels is needed to characterize the embrittlement potential of Mn-Ni-enriched precipitates (MNPs), particularly for the high fluence regime. While understanding of copper-enriched precipitates (CRPs) have been fully developed, the discovery and experimental verification [e.g., 4] of ‘late blooming’ MNPs with little to no copper for nucleation has stimulated research efforts to understand the evolution of these phases. New and existing databases will be combined to support development of physically based models of TTS for high fluence-low flux ($\phi < 10^{11}$ n/cm^2-s) conditions, beyond the existing surveillance database, to neutron fluences of at least 1×10^{20} n/cm^2 (>1 MeV). All references to neutron flux and fluence in this report are for fast neutrons (>1 MeV).

The RPV task of the LWRS Program is working with various organizations to obtain archival surveillance materials from commercial nuclear power plants to allow for comparisons of the irradiation-induced microstructural features from reactor surveillance materials with those from similar materials irradiated under high flux conditions in test reactors, such as the UCSB ATR-2 experiment. This report is submitted relative to the Milestone M3LW-15OR0402014 – “Complete Report Detailing Disassembly and Post-Irradiation Experiments for University of California Santa Barbara Advanced Test Reactor-2 Experiment.” Much of the text in the following sections is from a previous progress report [5] and is repeated here for the convenience of the reader.

2. BACKGROUND AND REVIEW OF UCSB ATR-2 EXPERIMENT

To obtain high fluence data in a reasonable time (e.g., ~ one or two years), test reactor experiments must be performed in such a way to enable development of a mechanistic understanding of the effects of flux [2, 3]. As described previously, such an irradiation experiment has been performed as part of the Idaho National Laboratory (INL) Advanced Test Reactor (ATR) National Scientific User Facility (NSUF). The experiment was awarded to University of California, Santa Barbara (UCSB) and its collaborator, ORNL, several years ago with full funding for the irradiation experiment in the ATR provided by DOE through the NSUF. A description of the UCSB ATR-2 experiment and materials was provided in previous progress reports [5, 6, 7] and also summarized in [8].

In collaboration with UCSB the INL staff carried out conceptual design of the sophisticated instrumented irradiation test assembly (capsule). The INL staff carried out the engineering design, construction and insertion of the test assembly, and was responsible for operation of the UCSB ATR-2 irradiation experiment. The scientific experiment itself was designed by UCSB in collaboration with ORNL. The total of 173 alloys included in the experiment were acquired by UCSB and ORNL, including those contributed by Rolls Royce Marine (UK), Bettis Atomic Power Laboratory (US), and the Central Research Institute for the Electric Power Industry (Japan). Notably, the Rolls Royce contribution included a total of more than 50 new alloys. Additionally, surveillance materials from various operating nuclear reactors were obtained from U.S. Nuclear Industry organizations with the assistance of Mr. William Server of ATI-Consulting and are included to enable a direct comparison of results from a test reactor at high flux and a power reactor at low flux. The specific surveillance materials were described in detail in [5, 6, 7] and are summarized in Section 3 of this report. Fabrication of the specimens was primarily carried out by UCSB with the assistance of ORNL. The specimens were loaded into 13 thin walled cups (cylindrical tubes) at UCSB and the cups were loaded into the test assembly at INL.

The irradiation was carried out in the so-called “Small I” position in ATR just inside the pressure vessel and reflector. The test assembly has a 20 mm inside diameter and is ≈ 1.2 m long. The UCSB ATR-2 experiment includes 1664 small specimens in three basic geometries. These include (1) tensile specimens, for a large matrix of alloys; (2) so-called multipurpose disc coupons (MPC) that will support microhardness, shear punch and a wide variety of microstructural characterization studies (e.g., small-angle neutron scattering, atom probe, etc) for all the alloys; (3) 20-mm diameter disc compact tension (DCT) fracture specimens for three alloys - the Palisades B weld and two UCSB forgings (C17 and LP). The test assembly included a gadolinium thermal neutron shield and active temperature control with three major regions at nominal temperatures of 270, 290 and 310°C, and one small region at 250°C. The gadolinium shield was incorporated to minimize activities of the specimens for PIE. The specimens were irradiated at a peak flux of about 3.3×10^{12} n/cm^2-s (>1 MeV) to a target fluence of 1×10^{20} n/cm^2. The identification, general specimen types, target irradiation temperature, and nominal target fluence (ϕt) for each of the 13 cups included in the ATR-2 capsule were provided in [8]. Thus, a variety of relatively small specimens of many different RPV steels have been incorporated, including many materials that have been irradiated and tested in previous test reactor programs at different flux levels.

3. DESCRIPTION OF MATERIALS AND SPECIMENS

A summary of the materials, specimen types and numbers is provided in Table 3.1, while the materials are described in greater detail in [8]. As mentioned earlier, 173 alloys with 1664 specimens are included in the capsule. The DCT matrix includes three alloys, the Palisades B weld and two UCSB forgings (C17 and LP), while the UCSB commercial alloys include HSST Plate 02, HSSI Weld 73W, Midland Beltline Weld (WF-70), and other alloys from the UCSB IVAR project, etc. Additionally, surveillance materials from various operating nuclear reactors, designated ORNL alloys in Table 3.1, are included and were identified as those that would provide results of particular interest to the ATR-2 experimental objectives. These materials were identified based not only on their chemical composition but also on their inclusion in capsules intended for relatively high fluence to allow for comparisons of results from surveillance conditions and the test reactor conditions in the ATR-2 and subsequent experiments. From the group of materials identified as potential candidates, and with the major assistance of ATI-Consulting, we were able to procure nine specific RPV surveillance materials for inclusion in the ATR-2 capsule and they are shown in Table 3.2.

Table 3. 1 Specimen Matrices Summary

	Lg Disc		Sm Disc		Tens		DCT		Any Type	
Total # alloy/irrad cond	Alloy	Spc.	Alloy	Spc.	Alloy	Spc.	Alloy	Spc.	Alloy	Spc.
Total # spc	144	1028	40	224	55	367	3	45	173	1664
DCT matrix	3	14			3	54	3	45	3	113
CM alloys	21	231			13	92			21	323
Laval alloys	10	72			8	48			10	120
UCSB Commercial alloys	13	107	1	4	9	53			13	164
EPRI alloys	20	141			6	21			20	162
ORNL alloys	5	64	5	41	8	51			9	156
RR alloys	57	356	11	80	8	48			68	484
Bettis alloys	5	25							5	25
CRIEPI alloys			13	65					13	65
OV model alloys	9	15	10	34					10	49
Diffusion Multiples	1	3							1	3

More detailed lists of alloys and specimens are shown in Appendix A and Appendix B of [8], with Appendix B indicating the irradiation temperatures for the various alloys and specimens. Additionally, Appendix C of [8] contains individual tables for the various groups of materials for easier reference by material group. In summary, a variety of relatively small specimens of many different RPV steels have been irradiated in UCSB ATR-2, including many materials that have been irradiated and tested in previous test reactor and surveillance programs at different flux levels.

Table 3.2. List of archival surveillance materials.

Plant	Material	Heat Number	Specimen Provided[1]
Farley Unit 2	SMAW	BOLA	One (1) 1/2T-CT **“CW25”**
Farley Unit 2	SA533B-1	C7466-1	Two (2) 1/2T-CT **“CT29”** and **“CL28”**(a)
V.C. Summer	Linde 124 Weld	4P4784	One (1) 1/2T-CT **“CW26”**
Kewaunee	Linde 1092 Weld	1P3571	0.5” x 3” x 1.5” slice of weldment (weld marked)
Maine Yankee	Linde 1092 Weld	1P3571	Two (2) untested tensile “**4KL”** and **“3J2”** Two (2) broken Charpy halves from specimen **“372”**
Farley Unit 1	Weld	33A277	
Beaver Valley Unit 2	Plate	B9004-1	Block 5×2.25×2.375 in.
Kewaunee	Forging, SA 508-2	B6307-1	Block 3.19×0.875×0.55 in.
Turkey Point Unit 4	Linde 80 Weld, SA-1094	Weld wire heat #71249 and Linde 80 flux lot 8457.	Block 3.375x4.25x8.625 in. (Block returned following machining of specimens)

Notes: [1] “CT” refers to transverse orientation and “CL” refers to longitudinal orientation.

Tensile specimens will be tested in accordance with ASTM Standard Test Procedure E8 to obtain yield and ultimate strengths for comparison with those of the unirradiated condition and with the microhardness and shear punch results. Those results will be correlated to ascertain the effects of irradiation on hardening. The excellent correlation between tensile specimen yield strength and shear punch strength measurements was shown in [8]. The DCT specimens will be tested in accordance with ASTM Standard Test Method E-1921 to obtain the reference temperature, T_0, for comparison with that of the unirradiated condition. A diffusion-multiple specimen was also designed and prepared by UCSB and is included in the capsule.

4. THE ATR-2 EXPERIMENT CAPSULE DISASSEMBLY

Fabrication and assembly of the UCSB ATR-2 irradiation test assembly was completed in late spring of 2011 and was successfully installed in the ATR on May 26, 2011. The irradiation began on June 7, 2011 and was anticipated to achieve its target fluence of 1×10^{20} n/cm^2 (E>1 MeV) in the autumn of 2012. A number of delays in operation of the ATR pushed the completion of the ATR-2 irradiation campaign to January of 2014 following completion of cycle 155A. Chief among these was the Powered Axial Locator Mechanism (PALM) cycle. Thermocouple monitors during the course of the irradiation campaign have shown that the specimens are generally being irradiated at or close to their target temperatures, but the final determination of irradiation temperatures and dosimetric information (i.e., neutron flux and fluence) for the test specimens are not yet available. The irradiation (ATR cycle 155A) was completed on 17 January 2014with peak and average fluences of 1.3 and 1.0×10^{20} n/cm^2 (>1 MeV), respectively, which meets the objective of reaching a fast fluence of at least 0.90×10^{20}n/cm^2. Following completion of the irradiation campaign, the ATR-2 capsule was stored in the ATR canal until ready for disassembly operations. In June of 2014, the experiment was cropped in the canal. A dry cap was installed and the experiment passed the 30 psi pressure test, while the bails on the capsule were successfully removed with an underwater saw specifically designed and fabricated for that purpose. In July, the experiment was moved into a cask suitable for internal transfers within INL, and in August the experiment was moved into the Dry Transfer Cubicle (DTC) for final sizing. In early October of 2014, the experiment was sized in the DTC following significant difficulty with cutting through some of the irradiation-hardened capsule material and then sent to the INL Materials and Fuels Complex (MFC) for subsequent disassembly to remove the 13 cups that contain the test specimens.[1]

The original concept of opening the ends of the capsule and pushing the 13 cups out of the capsule was met with great difficulty and was eventually abandoned in favor of using a mill to cut the capsule open longitudinally and remove the cups by prying open the capsule. This difficult operation was completed in mid-June of 2015 with some of the very thin-wall cups cut open by the mill cutter. Figures 4.1 and 4.2 show examples of an intact cup and one with the specimens spilled out of the cut open cup.[1] It appears at this time, however, that there was no damage that would compromise the utility of the specimens.

Following the disassembly operation described above, the specimen cups were loaded into transport pigs, placed in three shielded drums, and shipped to ORNL, where they were received on 10 July 2015 at the Irradiated Materials Examination and Testing (IMET) hot

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[1] The information in these paragraphs was gleaned from email messages sent primarily by Colin Knight and Ian Chestnut of the Idaho National Laboratory.

cell facility.

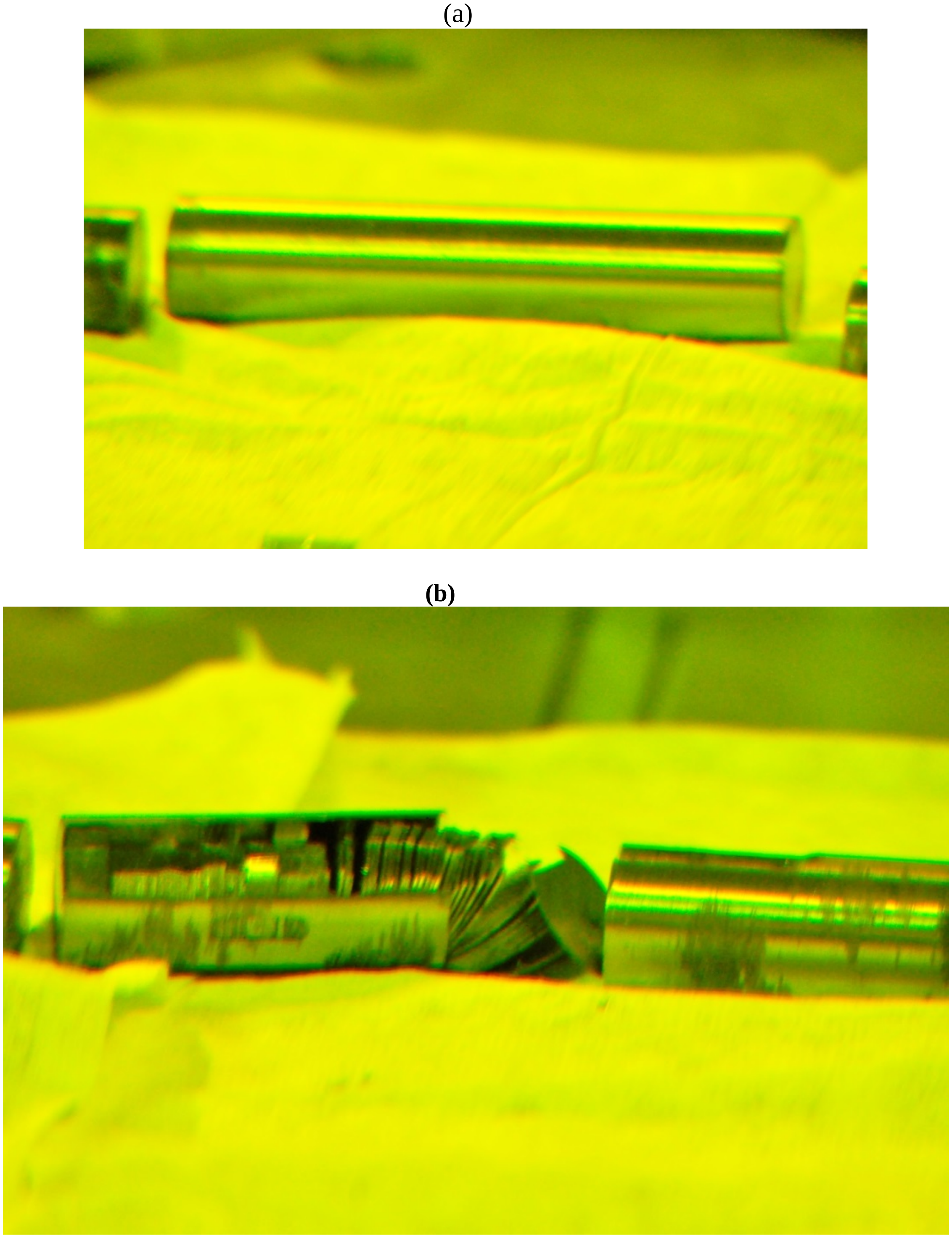

Figure 4.1. Photos of the UCSB ATR-2 experiment in the INL hot cell showing (a) a specimen cup intact, and (b) a specimen cup cut open with muti-purpose disc specimens spilling out of the cup.

5. POST-IRRADIATION EXAMINATION STATUS

The PIE plan for examination of the ATR-2 experiment is considered to be fluid and flexible to accommodate changing emphasis on results in order to meet project objectives. It was also not known with certainty that the radioactivity of the samples would allow for most of them to be tested in the ORNL Low Activated Materials Development and Analysis (LAMDA) laboratory and at UCSB. Activity measurements performed on selected specimens from cup 7 at the ORNL IMET hot cells confirmed that the activities of the specimens indicate that most have sufficiently low activity to enable testing in those low activity laboratories. The general steps for the PIE evaluation and priorities are discussed in detail in [5, 8]. Because the specimens arrived significantly later than the plan in [5, 8], the top priority was revised to gain retrieval of the drum and pig containing cup 7 which contains specific tensile specimens required for testing by UCSB. In mid-August, the drum containing cup 7 was moved into the IMET cells, the pig removed and the specimen cups retrieved. Cup 7 was then opened and the small disc-shaped boxes, TH4 and TH5, containing the tensile specimens were identified, moved to a different cell and opened to identify the tensile specimens. The tensile specimens were subsequently loaded into fiber tubes, loaded into pigs and prepared for shipping to UCSB. The tensile specimens were shipped to UCSB in mid-September. The microhardness test system purchased by ORNL for this program is in the process of being moved into the LAMDA laboratory; the system has been utilized by many userS in the meantime and shown to be a robust and accurate microhardness testing system.

Specific test sequences will be determined by priority guidelines as well as on final determination of irradiation temperatures and dosimetric data for the wide range of specimens and materials. The PIE activities are substantial due to the very large number of materials and specimens and will involve multiple collaborations with many organizations. Moreover, the availability of facilities and test equipment can easily require changes to the initial schedule in order to maximize productivity. Now that activity measurements have been made, cost estimates for the PIE are underway, including those for activities associated with CRIEPI, Bettis, Rolls-Royce, and EPRI (for the surveillance materials). The general steps for the PIE evaluation are as follows:

1. Selected cups, with priority given to cups 6, 7, and 8, will be opened to retrieve and identify individual specimens, with further packaging of like specimens by material/specimen type/irradiation temperature/fluence. **<u>As mentioned above, cup 7 has been opened and tensile specimens sent to UCSB.</u>**
2. Dosimeter packets will be retrieved for individual dosimeter identification and shipment for counting and analysis of the fluence for each dosimeter.
3. Activity measurements of individual specimen packets will be performed in preparation for shipment to other locations for testing, such as the LAMDA laboratory in Building 4508.
4. Small specimens will be punched from selected mutli-purpose discs and shipped to UCSB. Microhardness measurements will be used to complement mechanical testing at UCSB and at ORNL and to conduct mechanism experiments, including post-irradiation annealing studies based on microhardness testing. The annealing studies will also provide a basis to develop remediation annealing and reirradiation models. The small specimens will also be used for extensive characterization studies, including APT, TEM, SANS, and other microstructural and microanalytical evaluations. Additionally, a selected number of MPCs will be shipped to UCSB for initial shear punch testing to validate the UCSB-designed shear punch system prior to its installation in the LAMDA laboratory at ORNL.
5. However, the majority of mechanical property tests will be performed at ORNL. Ultimately, the plan is to perform shear punch tests on a majority of the alloy/irradiation conditions at ORNL, which will take advantage of the automated shear punch instrument developed by UCSB.

6. The plan is also to perform redundant room temperature tests on the tensile specimens for all corresponding alloy/irradiation conditions.
7. The compact tension specimens will be tested to establish transition temperature shifts and provide additional insight on fracture toughness Master Curve methodology.
8. Testing of the specimens will be performed in accordance with the standard practices where applicable.

The nominal order of priority regarding testing of the various materials is as follows:

1. In the case of the MPC and tensile specimen testing, a preliminary priority sequence of testing will be on the high fluence (290°C) irradiation condition as follows:
    a. Tensile tests of UCSB forgings LB, LC, LD, LG, LH, LI, LJ, C6, C9, C31, and WA. **These specimens from tensile holders TH4 and TH5 in cup 7 have been retrieved and shipped to UCSB.**
    b. ORNL surveillance materials, **Retrieval of these specimens is in progress.**
    c. UCSB and Rolls-Royce alloys that have been irradiated over a wide range of flux in IVAR and other experiments,
    d. The Rolls-Royce matrix of new alloys that explore extended RPV compositional space,
    e. Selected EPRI, CRIEPI and Bettis alloys that specifically complement the matrix of tests cited in a through c above,
    f. Additional tests on other alloys.
2. For MPC and tensile testing, a selected subset of key alloys will be characterized at the lower fluences and at the lower and higher irradiation temperatures in order to establish the hardening dependencies on these variables. The matrix will include the down-selected alloys from items 1a through 1c. Establishing these dependencies will have an equal priority with the compositional-based assessments in item 1 above.
3. The compact tension specimens for each material will be tested in accordance with ASTM E1921 at a minimum of three temperatures to provide ΔT_0 shifts and information on possible changes in Master Curve shape for highly embrittled materials.
4. Additional mechanical property tests will be conducted on a subset of alloys to complement the mechanistically oriented annealing experiments at UCSB and provide a basis for developing remediation annealing models.
5. Extensive microstructural/microanalytical characterization studies using a variety of complementary techniques will be conducted based on mechanistic insight that identified gaps and as guided by the mechanical property test results.

The following is an example of the sequence of measurements that would be made on a specific high priority alloy for various irradiation conditions. This specific example is developed for the Palisades RPV weld:

1. Shear punch, tensile and microhardness measurements to establish the changes in yield strength and post-yield constitutive properties for a matrix of irradiation fluence and temperature conditions;
2. Fracture toughness tests to provide ΔT_0 shifts and information on possible changes in Master Curve shape for highly embrittled materials and to relate the ΔT_0 shifts to

changes in yield strength. The fracture analysis on this alloy will include fractography and micromechanical evaluations to inform fracture modeling of Master Curve issues;

3. The as-irradiated conditions will be characterized by APT, TEM, SANS and PAS;
4. The material will be subject to a series of post-irradiation annealing treatments followed by microhardness tests, shear punch tests and microstructural and microanalytical characterizations.

An approximate timeline for the testing and evaluations discussed above is shown in Appendix A, which has been revised to reflect the later arrival of the specimens at ORNL relative to that shown in [8].

6. SUMMARY

The primary objective of the LWRSP RPV task is to develop robust predictions of transition temperature shifts (TTS) at high fluence (ϕt) to at least 10^{20} n/cm^2 (>1 MeV) pertinent to plant operation of some pressurized water reactors (PWR) for 80 full power years. The RPV task of the LWRS Program is working with various organizations to obtain archival surveillance materials from commercial nuclear power plants to allow for comparisons of the irradiation-induced microstructural features from reactor surveillance materials with those from similar materials irradiated under high flux conditions in test reactors. Additionally, the task is collaborating and cooperating with the University of California Santa Barbara regarding post-irradiation examination of the materials and specimens in the ATR-2 experiment. The ATR-2 capsule completed irradiation on 17 January 2014 with the average and peak fluences at the end of cycle 155A of 1.0 and 1.3×10^{19} n/cm^2, respectively. The capsule was disassembled at INL and 13 specimen cups were shipped to ORNL with arrival at the Irradiated Materials Examination and Testing facility hot cells on 10 July 2015. This report has summarized the experiment, a detailed description of the materials and test specimens, the post-irradiation activities completed to date (e.g., shipment of specific tensile specimens to UCSB), and the plan for continued testing and examination of the irradiated specimens.

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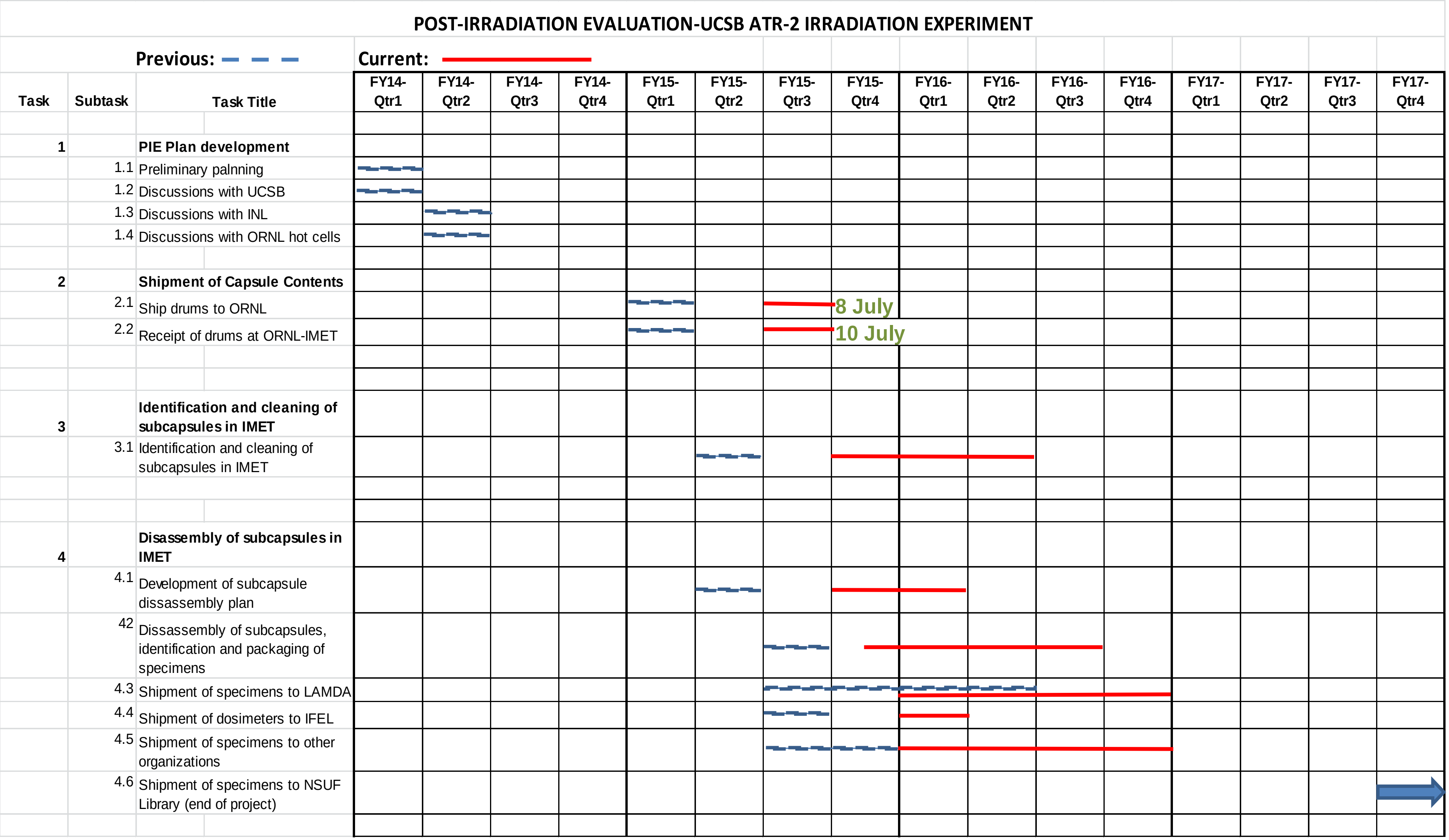
APPENDIX A. REVISED SCHEDULE FOR POST-IRRADIATION EXAMINATION OF ATR-2 EXPERIMENT.

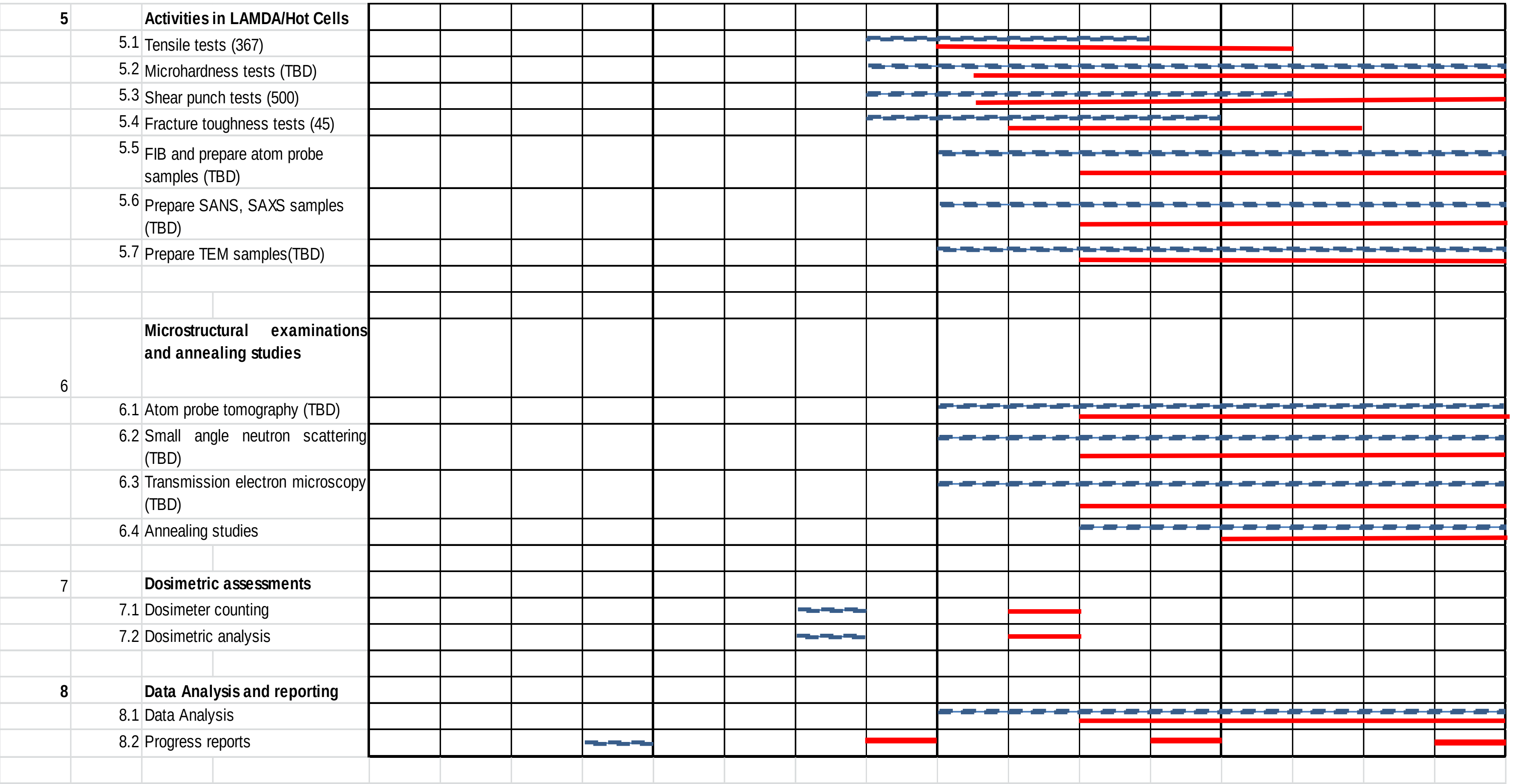


POST-IRRADIATION EVALUATION-UCSB ATR-2 IRRADIATION EXPERIMENT

Previous: – – – **Current:** ——

Task	Subtask	Task Title	FY14-Qtr1	FY14-Qtr2	FY14-Qtr3	FY14-Qtr4	FY15-Qtr1	FY15-Qtr2	FY15-Qtr3	FY15-Qtr4	FY16-Qtr1	FY16-Qtr2	FY16-Qtr3	FY16-Qtr4	FY17-Qtr1	FY17-Qtr2	FY17-Qtr3	FY17-Qtr4
1		**PIE Plan development**																
	1.1	Preliminary palnning																
	1.2	Discussions with UCSB																
	1.3	Discussions with INL																
	1.4	Discussions with ORNL hot cells																
2		**Shipment of Capsule Contents**																
	2.1	Ship drums to ORNL								8 July								
	2.2	Receipt of drums at ORNL-IMET								10 July								
3		**Identification and cleaning of subcapsules in IMET**																
	3.1	Identification and cleaning of subcapsules in IMET																
4		**Disassembly of subcapsules in IMET**																
	4.1	Development of subcapsule dissassembly plan																
	42	Dissassembly of subcapsules, identification and packaging of specimens																
	4.3	Shipment of specimens to LAMDA																
	4.4	Shipment of dosimeters to IFEL																
	4.5	Shipment of specimens to other organizations																
	4.6	Shipment of specimens to NSUF Library (end of project)																

APPENDIX A. PRELIMINARY SCHEDULE FOR POST-IRRADIATION EXAMINATION OF ATR-2 EXPERIMENT (CONT’D).



5		**Activities in LAMDA/Hot Cells**
	5.1	Tensile tests (367)
	5.2	Microhardness tests (TBD)
	5.3	Shear punch tests (500)
	5.4	Fracture toughness tests (45)
	5.5	FIB and prepare atom probe samples (TBD)
	5.6	Prepare SANS, SAXS samples (TBD)
	5.7	Prepare TEM samples(TBD)
6		**Microstructural examinations and annealing studies**
	6.1	Atom probe tomography (TBD)
	6.2	Small angle neutron scattering (TBD)
	6.3	Transmission electron microscopy (TBD)
	6.4	Annealing studies
7		**Dosimetric assessments**
	7.1	Dosimeter counting
	7.2	Dosimetric analysis
8		**Data Analysis and reporting**
	8.1	Data Analysis
	8.2	Progress reports