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Identification and Shipment Progress of The INL and LANL Samples to Be Received at ORNL

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Light Water Reactor Sustainability (LWRS)

IDENTIFICATION AND SHIPMENT PROGRESS FOR THE INL AND LANL SAMPLES TO BE RECEIVED AT ORNL

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EXCUTIVE SUMMARY

Ferritic-martensitic steel Grade 92, with T91 as a reference, together with austenitic stainless steel (or Incoloy Alloy) 800H and its grain-boundary-engineered version 800H-TMP (ThermoMechanical Processing), is investigated in this project. A total of fifteen Grade 92 samples from two or three heats, four T91 samples from two heats, and six 800H and six 800H-TMP samples from one heat were selected, which were primarily irradiated in the Advanced Test Reactor of Idaho National Laboratory (INL), with the rest in the High Flux Isotope Reactor of Oak Ridge National Laboratory (ORNL), BOR-60 of Russia, and Phénix reactor of France. The samples were irradiated in the temperature range of 241 to 720°C and a dose range of 1.28 to ~70 displacements per atom.

All the INL samples, most of the ORNL samples, and part of the LANL (Los Alamos National Laboratory) samples from the ATR, BOR-60 and Phénix reactors have been received at the Irradiated Materials Examination and Testing (IMET) hot cell facility and Low Activation Materials Design and Analysis (LAMDA) laboratory of ORNL. Post-irradiation examination of the received samples is in progress at ORNL. The effects of irradiation temperature, dose, reactor (e.g., dose rate, neutron-spectrum-induced difference in transmutation), TMP, and heats variation on the microstructures and mechanical properties of the samples will be evaluated.

1. INTRODUCTION

Advanced alloys are desired to provide greater safety margins, design flexibility and economics compared to traditional reactor materials. Grade 92 ferritic-martensitic steel and austenitic Alloy 800/800H are two of the promising alloys interested by the current Advanced Radiation-Resistant Materials (ARRM) and Light Water Reactor Sustainability (LWRS) programs. However, systematic studies on neutron-irradiation induced changes in microstructures and mechanical properties are deficient for the alloys. The objective of this project is to develop correlations between microstructures and mechanical properties of the neutron-irradiated Grade 92 and Alloy 800/800H, based on the experimental results generated from this work. It is expected to develop broader correlations for these types of steels by comparing the results of this work with that of similar alloys such as Grade 91, Alloy 709 and type 304/316 stainless steels from literature and the ongoing studies, with the aid of thermodynamics, kinetics, and microstructural hardening modeling.

Samples of Grade 92 and Alloy 800H selected in this work were primarily irradiated in two test reactors for up to ~14 displacements per atom (dpa) at ~241–720°C. Samples of Grade 91, irradiated in the same reactors, were selected as references of Grade 92. Few samples from other two reactors will be included for comparison. Both irradiated and unirradiated samples from the same heat of the materials will be examined to elucidate the radiation-induced evolutions in microstructures, mechanical properties, and deformation mechanisms. To be more specific, mechanical properties such as tensile properties, modulus, hardness, and viscoplasticity will be measured through tensile, Vickers hardness and nanoindentation tests. Microstructural characterization of the samples will be carried out using the state-of-the-art instruments and techniques provided through the Nuclear Science User Facilities (NSUF). The obtained experimental results will then be used to establish the knowledgebase on the effects of alloy chemistry, thermomechanical-processing, and irradiation conditions on microstructures and mechanical properties of Grade 92 and Alloy 800H.

Outcomes of this project will include a comprehensive set of data including microstructures and mechanical properties of both irradiated and unirradiated samples of the interested steels, which will not only help understanding the essential performance of similar alloys, but more importantly to gain indispensable insights into the development of advanced alloys with superior radiation resistance. The outcomes can also serve as inputs and/or benchmarks for microstructural and mechanical property modeling of irradiated ferritic-martensitic and austenitic steels. The accomplishment of this project will directly benefit the LWRS program and bring values to the Advanced Reactor Technologies and Small Modular Reactors programs.

Procurement of the interested neutron-irradiated samples is a critical step to accomplish the goal of this project. This report summarizes the identification and shipment progress for the interested samples at Idaho National Laboratory (INL), Los Alamos National Laboratory (LANL), and Oak Ridge National Laboratory (ORNL), which are to be received and examined at ORNL for this project.

2. INL SAMPLES

2.1 SELECTED INL SAMPLES

A total of twelve INL samples of steels 800H, NF616, and T91 were selected, which were irradiated in the Advanced Test Reactor (ATR) of INL through the University of Wisconsin Pilot Project of the ATR National Scientific User Facility [1]. The steels 800H, NF616, and T91 were commercial heats distributed by INL, Japan Atomic Energy Agency (JAEA), and INL, respectively. The selected samples are listed in Table 1. The samples are in two types, with one type as type SS-J2 miniature specimens and the other type as 3-mm diameter discs.

Table 1. Selected INL samples.

Specimen type	Alloy	Engraved sample code	KGT Num	Temperature (°C)		Dose (dpa)		Dose rate (dpa/s)
				Planned	Average as-run	Planned	As-run total	
Type SS-J2 miniature tensile specimen with 16 × 4 × (<1) mm and gauge 5 × 1.2 × (<1) mm.	800H	N4	1712	400	359	6	7.27	1.30×10^{-7}
		N5	1772	500	451.5	3	3.9	1.35×10^{-7}
		N6	1806	500	431	6	9.01	1.61×10^{-7}
	800H-TMP	P4	2578	400	359	6	7.36	1.31×10^{-7}
		P5	2596	500	451.5	3	3.95	1.37×10^{-7}
		P6	2597	500	431	6	9.12	1.63×10^{-7}
	NF616	D1*	402	300	241	3	3.51	1.29×10^{-7}
		D2	1791	300	291.5	6	2.96	5.29×10^{-8}
		D4	1735	400	359	6	5.91	1.06×10^{-7}
		D6	1783	500	431	6	8.16	1.46×10^{-7}
3-mm diameter disc (~0.2-mm thick)	T91	A4	1729	400	447.5	6	4.78	8.51×10^{-8}
		A6	1790	500	429.5	6	7.79	1.39×10^{-7}

* The sample D1 is at Argonne National Laboratory (ANL), which is to be in-situ tensile-tested during high-energy x-ray diffraction using the Advanced Photon Source (APS) of ANL.

Other than the standard solution-annealed condition (1177°C for 24 minutes per centimeter of thickness, followed by a water quench) of austenitic stainless steel 800H, a thermomechanically processed (TMP) condition, named as 800H-TMP, was irradiated simultaneously. The TMP was constituted of a 6.6 ± 0.2 % thickness reduction by rolling at room temperature, followed by annealing at 1050°C for 90 minutes and water quench, which is a grain boundary engineering (GBE) method to significantly increase the fraction of low-Σ coincidence site lattice (CSL) boundaries, e.g., nearly 70% (800H-TMP) and ~40% (800H) low-Σ CSL boundaries [2]. GBE with a significantly increased fraction of low-Σ CSL boundaries would benefit a variety of properties such as strength and resistance to creep, stress corrosion cracking, and oxidation [3]. The 800H-TMP exhibited noticeable enhancements in the resistance to thermal aging [4] and corrosion in supercritical water and high-temperature air [5,6,7,8]. Preliminary studies also showed more or less improvements in resistance to neutron irradiation [9,10]. Therefore, three samples of 800H-TMP, together with three samples of 800H irradiated in nearly identical conditions, were selected in this project to confirm and elucidate the beneficial effects of GBE/TMP on the neutron irradiation resistance of 800H. The other set of samples are ferritic-martensitic steels NF616 and T91, classic/typical versions of Grade 92 and 91, respectively. T91 samples are to be used as reference for NF616.

Each sample was engraved with a unique sample code for visual sample identification and assigned with a unique KGT number for sample library record. The information, together with the planned and as-run irradiation temperature and dose listed in Table 1, was obtained from the material library presented online at https://nsuf.inl.gov. The dose rate in Table 1 was deduced from as-run neutron fluence divided by irradiation time. The irradiation conditions, e.g., temperature and dose, will be compared with the analytical reports of the University of Wisconsin Pilot Project [11,12,13].

The compositions in weight percent (wt%) of 800H, NF616, and T91 of the INL samples are listed in Table 2 [1]. The compositions are critical for alloy thermodynamic analysis and transmutation analysis to interpret the experimental observations in this project.

Table 2. Compositions in weight percent (wt%) of the INL samples, with Fe as balance*.

Alloy	Cr	Ni	Mn	Si	Ti	Al	V	W	Mo	Nb	Cu	C	N	P	S
800H/800-TMP	20.42	31.59	0.76	0.13	0.57	0.50					0.42	0.069		0.014	0.001
NF616	8.82	0.174	0.45	0.102		0.005	0.194	1.87	0.468	0.064		0.109	0.0474	0.012	0.0032
T91	8.37	0.21	0.45	0.28		0.022	0.216		0.90	0.076	0.17	0.1	0.048	0.009	0.003

* The blank cells are the elements not measured or reported. Oxygen and boron contents were reported as 0.0042% and 0.0017%, respectively, in NF616, which were not reported in the other alloys. 800H was solution-annealed at 1177°C for 24 minutes per centimeter of thickness, followed by a water quench. 800H-TMP was based on 800H, subjected to ~6.6% thickness reduction by rolling at room temperature and then annealed at 1050°C for 1.5 h with water quench. NF616 was normalized at 1070°C for 2 h and tempered at 770°C for 2 h with air cooling. T91 was normalized at 1066°C for 0,8 h and tempered at 790°C for 0.7 h with air cooling.

2.2 RECEPTION OF THE INL SAMPLES AT ORNL

One of the NF616 samples, with an engraved sample code of D1, is at ANL, which will be examined by in-situ tensile test during high-energy x-ray diffraction using the APS of ANL. The tested D1 will be shipped to ORNL for microstructural characterization and hardness measurements. Therefore, a total of eleven samples, i.e., nine SS-J2 tensile specimens and two 3-mm diameter discs, were shipped from INL to ORNL. ORNL has received the eleven samples in hot cells at 3025E. The nine SS-J2 tensile specimens are planned to be tensile-tested at room temperature in next few months right after the completion of instrument updates.

3. LANL SAMPLES

3.1 SELECTED LANL SAMPLES

Three sets of LANL samples were identified, which are classified by their irradiation reactors, i.e., ATR, BOR-60, and Phénix. The samples and their respective conditions are listed in Table 3, together with their alloy compositions listed in Table 4.

Table 3. Selected LANL samples.

<table>
<tr><th>Specimen type</th><th>Alloy</th><th>Sample ID</th><th>Temperature (°C)</th><th>Dose (dpa)</th><th>Irradiation reactor</th><th>Comment</th></tr>
<tr><td rowspan="2">Type SS-J2 tensile</td><td rowspan="3">T91</td><td>TA04</td><td>295</td><td>6.5</td><td rowspan="2">ATR</td><td rowspan="2">Tensile-tested at 25°C [14]</td></tr>
<tr><td>TA#1c [a]</td><td></td><td>0</td></tr>
<tr><td rowspan="4">Ø3-mm TEM discs</td><td>T108</td><td>517</td><td>28</td><td rowspan="4">BOR-60</td><td rowspan="4">Shared with an IRP project</td></tr>
<tr><td rowspan="3">NF616</td><td>N71</td><td>425</td><td>19.6</td></tr>
<tr><td>N64</td><td>524</td><td>15.4</td></tr>
<tr><td>N133</td><td>517</td><td>28</td></tr>
<tr><td rowspan="3">Type SS-J3 tensile or Ø3-mm TEM discs</td><td>800H</td><td rowspan="3">TBI [b]</td><td rowspan="3">~400 and ~500 [c]</td><td rowspan="3">Up to ~70 [c]</td><td rowspan="3">Phénix</td><td rowspan="3">The Phénix MATRIX experiment</td></tr>
<tr><td>800H-TMP</td></tr>
<tr><td>NF616</td></tr>
</table>

[a] Unirradiated control sample of the irradiated T91 (e.g., TA04).
[b] TBI – to be identified.
[c] Planned temperatures and dose, unlike the other analyzed temperatures and doses.

Table 4. Compositions in weight percent (wt%) of the LANL samples, with Fe as balance.

<table>
<tr><th>Alloy</th><th>Cr</th><th>Ni</th><th>Mn</th><th>Si</th><th>Ti</th><th>Al</th><th>V</th><th>W</th><th>Mo</th><th>Nb</th><th>Cu</th><th>C</th><th>N</th><th>P</th><th>S</th></tr>
<tr><td>T91 *</td><td>9.22</td><td>0.18</td><td>0.46</td><td>0.24</td><td>0.002</td><td>0.009</td><td>0.24</td><td>0.013</td><td>0.96</td><td>0.063</td><td>0.087</td><td>0.052</td><td>0.057</td><td>0.016</td><td>0.001</td></tr>
<tr><td>NF616</td><td colspan="15">The Phénix samples are from the same heat as the INL samples; the BOR-60 samples are likely to be the same heat as the INL samples, which is to be confirmed.</td></tr>
<tr><td>800H/800-TMP</td><td colspan="15">The Phénix samples are from the same heat as the INL samples.</td></tr>
</table>

* Also reported 0.002O and 0.021Co. The steel was normalized at 1040°C for 1 h with air cooling and tempered at 760°C for 1 h with air cooling [14]. The T91 sample T108 is from the same provider as that of the INL samples, which is likely to be the same heat to be confirmed.

The selected ATR set of samples only have one T91 sample (i.e., TA04 in Table 3) irradiated at ~295°C to ~6.5 dpa, which has the lowest irradiation temperature in all the identified T91 samples. It complements the INL T91 samples listed in Table 1 as a baseline for comparison with NF616. The TA#1c is an unirradiated control sample for TA04. The TA04 and TA#1c samples were tensile-tested at room temperature, with the results reported by Maloy et al. [14].

The selected BOR-60 set of samples have T91 and NF616 samples irradiated to 15.4–28 dpa at 425–524°C, which are shared with an Integrated Research Project (IRP) under the Nuclear Energy University Program (NEUP) of U.S. Department of Energy. The NF616 was from the same source as that in the INL samples. They are likely to be the same heat, which is to be confirmed. The BOR-60 samples also include 800H samples, which had been sufficiently characterized. The results will be included in this project for comparison. The BOR-60 samples are all TEM disc type specimens and thus only provide hardness and microstructure results. There are multiple samples per condition for each alloy. Therefore, the sample ID of the BOR-60 samples characterized in this project may be different from that listed in Table 3.

The selected Phénix set of samples include 800H, 800H-TMP, and NF616 samples irradiated up to ~70 dpa at planned irradiation temperatures of ~400 and ~500°C. The analyzed irradiation temperatures and doses will be available later. The samples are in type SS-J3 miniature tensile specimens (similar to SS-J2 with a thicker thickness) and 3-mm diameter TEM discs, which are from the same heat as that in the INL samples.

3.2 RECEPTION OF THE LANL SAMPLES AT ORNL

The ATR and BOR-60 sets of samples are being shipped to ORNL. The Phénix set of samples are expected to be available in early FY 2021, which may only provide some preliminary results for this project because of the timeline.

4. ORNL SAMPLES

4.1 SELECTED ORNL SAMPLES

The selected ORNL samples include G92-2b and 800H samples irradiated to 0.46–14.66 dpa at 400 to ~720°C in the High Flux Isotope Reactor (HFIR) of ORNL, which are listed in Table 5, together with the alloy composition and condition in Table 6. The G92-2b is a heat of optimized Grade 92, which was developed under the Advanced Reactor Technologies (ART) program [15]. It showed improved strength and creep resistance compared with conventional Grade 92 such as NF616. The AR2 and HG1 samples are 800H and 800H-TMP, respectively, from the same heat as the INL samples, as well as the Phénix set of samples, with composition listed in Table 2.

Table 5. Selected ORNL samples.

Specimen type	Alloy	Sample ID	Temperature (°C)	Dose (dpa)
SS-J2	G92-2b	GB03	400	0.52
		GB04	~460	7.44
		GB05	496.7	14.66
		GB10	683.3	0.46
		GB11	~720	7.44
		GB12	~720	14.63
SS-3	800H	AR2	580	1.28
	800H-TMP	HG1		

Table 6. Compositions in weight percent (wt%) of the ORNL samples, with Fe as balance.

Alloy	Cr	Ni	Mn	Si	V	W	Mo	Nb	C	N	B
G92-2b *	8.9	0.10	0.47	0.14	0.23	1.9	0.43	0.11	0.087	0.045	<0.002
800H/800-TMP	The same as the INL and Phénix samples.										

* The heat was normalized at 1080°C for 1 h, followed by hot-rolling to 0.6”-thick plate from 1” at 1080°C and water quench, and tempered at 750°C for 2 h with air cooling.

4.2 RECEPTION OF THE ORNL SAMPLES

The selected ORNL samples have been located. Some of the samples have been tensile-tested at the Irradiated Materials Examination and Testing (IMET) hot cell facility and being examined in the Low Activation Materials Design and Analysis (LAMDA) laboratory of ORNL, which is reported in a separate milestone report.

5. SUMMARY

Ferritic-martensitic steel Grade 92, with T91 as a reference, together with austenitic stainless steel (or Incoloy Alloy) 800H and its GBE-treated version 800H-TMP, is investigated in this project. A total of fifteen Grade 92 samples, four T91 samples, six 800H and six 800H-TMP samples were selected, which were primarily irradiated in the ATR, with the rest in the HFIR, BOR-60 and Phénix reactors. The irradiation temperature and dose conditions of the selected samples are plotted in Figure 1. The Grade 92 samples have a large irradiation condition range, with the T91 samples consistent in a couple of the conditions. The 800H and 800H-TMP samples have excellent consistency in the irradiation conditions. The selected samples of Grade 92 were from two or three heats, T91 from two heats, and 800H and 800H-TMP from one heat.

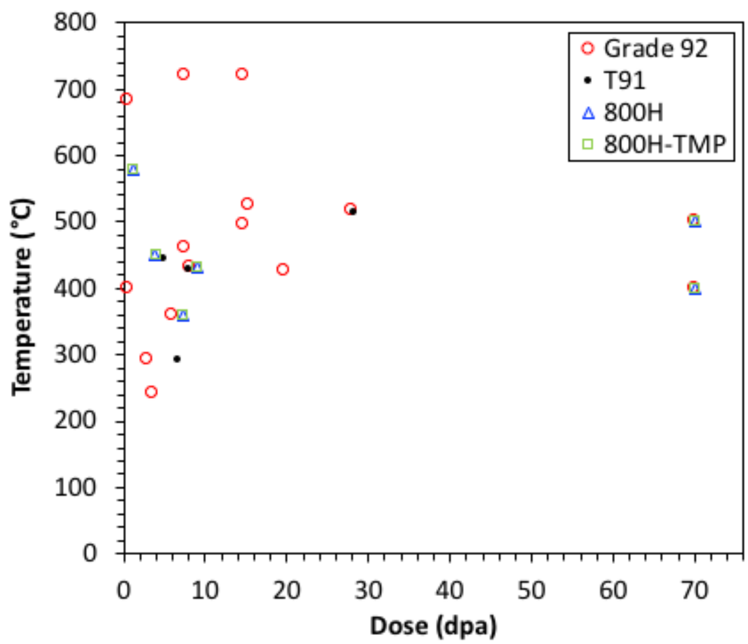

Figure 1. Irradiation temperature and dose conditions of the selected Grade 92, T91, 800H, and 800H-TMP samples.

All the INL samples, most of the ORNL samples, and part of the LANL samples have been received at the IMET hot cell facility and the LAMDA laboratory of ORNL. Post-irradiation examination of the received samples is in progress at ORNL. The effects of irradiation temperature, dose, reactor (e.g., dose rate, neutron-spectrum-induced difference in transmutation), TMP, and heats variation on the microstructures and mechanical properties of the samples will be evaluated.

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