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OAK RIDGE NATIONAL LABORATORY
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Light Water Reactor Sustainability Program:

Report on Status of Shipment of High Fluence Austenitic Steel Samples for Characterization and Stress Corrosion Crack Testing

Milestone M3LW-16OR0402024

September 2016

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Report on Status of Shipment of High Fluence Austenitic Steel Samples for Characterization and Stress Corrosion Crack Testing

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Executive Summary

The goal of the Mechanisms of Irradiation Assisted Stress Corrosion Cracking (IASCC) task in the LWRS Program is to conduct experimental research into understanding how multiple variables influence the crack initiation and crack growth in materials subjected to stress under corrosive conditions. This includes understanding the influences of alloy composition, radiation condition, water chemistry and metallurgical starting condition (i.e., previous cold work or heat treatments and the resulting microstructure) has on the behavior of materials. Testing involves crack initiation and growth testing on irradiated specimens of single-variable alloys in simulated Light Water Reactor (LWR) environments, tensile testing, hardness testing, microstructural and microchemical analysis, and detailed efforts to characterize localized deformation. Combined, these single-variable experiments will provide mechanistic understanding that can be used to identify key operational variables to mitigate or control IASCC, optimize inspection and maintenance schedules to the most susceptible materials/locations, and, in the long-term, design IASCC-resistant materials.

In support of this research, efforts are currently underway to arrange shipment of “free” high fluence austenitic alloys available through Électricité de France (EDF) for post irradiation testing at the Oak Ridge National Laboratory (ORNL) and IASCC testing at the University of Michigan. These high fluence materials, ranging in damage values from 45 to 125 displacements per atom (dpa), also have significant importance across multiple LWRS work packages. These high fluence samples will provide material for model validation in the Swelling (LW-16OR040204), Thermodynamic Tools for Evaluation of Radiation Effects (LW16OR040217), and Irradiation-Induced Phase Transformations in High-Fluence Core Internals (LW-16OR040205) work packages.

The samples identified for transport to the United States, which include nine, no-cost, 304, 308 and 316 tensile bars, were relocated from the Research Institute of Atomic Reactors (RIAR) in Dimitrovgrad, Ulyanovsk Oblast, Russia, and received at the Halden Reactor in Halden, Norway, on August 23, 2016. ORNL has been notified that a significant amount of work is required to prepare the samples for further shipment to Oak Ridge, Tennessee. The preliminary work for sample shipment between Halden and Oak Ridge includes fabrication of an inner cask sample container, decontamination and preparation of a Type A container, preparation of new activity calculations, all necessary paperwork, and handling. ORNL will continue to work closely with Faiza Sefta of EDF and Torill Karlsen and Helge Valseth of the Institute for Energy (IFE) in Norway to track progress of sample preparation and shipment status, and to work toward an agreement that covers material shipping costs between the Halden Reactor and the Oak Ridge National Laboratory.

I. Introduction

In support of Mechanisms of Irradiation Assisted Stress Corrosion Cracking (IASCC) research task within the Light Water Reactor Sustainability (LWRS) Program efforts are underway to arrange shipment of high fluence austenitic alloys available through Électricité de France (EDF) for post irradiation testing at the Oak Ridge National Laboratory (ORNL) and IASCC initiation testing at the University of Michigan. Nine high displacements per atom (dpa) specimens were originally identified by Jeremy Busby (ORNL), Gary Was (University of Michigan), and Faiza Sefta (EDF). These BOR-60 irradiated samples, which have recently been relocated from the Research Institute of Atomic Reactors (RIAR) in Dimitrovgrad, Ulyanovsk Oblast, Russia, to the Halden Reactor in Norway are identified in Table 1. The shipment from RIAR to Halden included samples for IASCC testing at Halden as part of a proposed Electric Power Research Institute (EPRI) program as well as additional samples that have been assigned either to the Halden or ORNL. The total number of samples expected at ORNL for the LWRS sponsored work increased to 16 and includes additional lower fluence 304 and 316 grade stainless steel samples that broaden the range of available testing. The additional samples also include alloy 308 steel samples. Figures of the sample design for both EPRI and LWRS projects follows. These samples are the high fluence companion samples to lower fluence (<45 dpa) materials that have formed the basis for much of the testing in the LWRS program at both ORNL and the University of Michigan [1-4].

Irradiated Specimens

Thirty-six (36) tensile specimens were transported from RIAR and received at Halden on August 23, 2016. These specimens are listed in Table 1 and are represented according to the color key immediately following the list. The nine samples identified for transport to the United States, highlighted purple, are tensile bar type GI-116 geometries shown in Figure 1. The shipment of material from RIAR to Halden also contains sub-size compact tension (CT) specimens and pressurized tube samples, highlighted blue, which will be utilized for in-reactor irradiation tests at Halden. In the table below, the E suffix stands for écroui, or loosely translating to mean hardened, as in through cold working. The suffix H represents the traditional solution annealed (SA) condition.

Item	Irradiation Program	Stage	Specimen Location	Heat	Nature of the Specimen	Dose (dpa)
1	Samara II	Stage 6	A76	304-1 H	Tensile	3.8
2	Samara II	Stage 1	B83	316-1 E	Tensile Hf	4.4
3	Samara I	Stage 1	B63	316-1 E	Tensile Hf	4.8
4	Samara II	Stage 5	B77	316-1 E	Tensile	4.9
5	Boris 6R 1-2	Stage 6	A109	304-1 H	Tensile	11.8
6	Boris 2	Stage 2	A40	304-1 H	Tensile	19.7
7	Boris 2	Stage 2	A41	304-1 H	Tensile	19.7
8	Boris 4	Stage 5	B108	316-1 E	Tensile	22.7
9	Boris 5	Stage 4	B9X	316-1 E	Tensile	25.4
10	Boris 4	Stage 4	A90	304-1 H	Tensile	42
11	Boris 4	Stage 4	A91	304-1 H	Tensile	42
12	Boris 6R 2-1	Stage 6	A35	304 H	Tensile	11.8
13	Samara II	Stage 1	A82	304-1 H	Tensile Hf	4

14	Samara		A56	304-H	Tensile Hf	5
15	Boris 5	Stage 7	SB24	308-2	Tensile	19.1
16	Boris 5	Stage 7	SB25	308-2	Tensile	19.1
17	Boris 4	Stage 4	A92	304-1 H	Tensile	42
18	Samara II	Stage 2	A81	304-1 H	Tensile Hf	4.4
19	Samara II	Stage 2	A80	304-1 H	Tensile Hf	4.5
20	Samara II	Stage 2	B81	316-1 E	Tensile Hf	5
21	Boris 2	Stage 2	SA5	308-1	Tensile	19.7
22	Boris 2	Stage 2	SA6	308-1	Tensile	19.7
23	Boris 5	Stage 4	A27	304-1	Tensile	69
24	Boris 6R 1-3	Stage 6	A84	304-1 H	Tensile	5.4
25	Boris 6R 1-2	Stage 6	A107	304-1 H	Tensile	11.8
26	Boris 2	Stage 3	A37	304-1 H	Tensile	21.7
27	Boris 5	Stage 3	A98	304-1 H	Tensile	69
28	Boris 6R 2-1	Stage 3	A96	304-1 H	Tensile	95
29	Boris 6R 2-1	Stage 1	A32	304-1 H	Tensile	125.4
30	Boris 4	Stage 4	B98	316-1 E	Tensile	42
31	Boris 5	Stage 5	B101	316-1 E	Tensile	46.9
32	Boris 5	Stage 5	B102	316-1 E	Tensile	46.9
33	Boris 5	Stage 5	B103	316-1 E	Tensile	46.9
34	Boris 5	Stage 4	B89	316-1 E	Tensile	67.4
35	Boris 5	Stage 4	B95	316-1 E	Tensile	67.4
36	Boris 5	Stage 1	B35	316-1 E	Tensile	125.4

Additional samples for Halden tests
Originally planned Halden samples
Additional samples available for LWRS testing
Originally planned LWRS samples

Table 1. Tensile specimens received from RIAR.

Sample Design

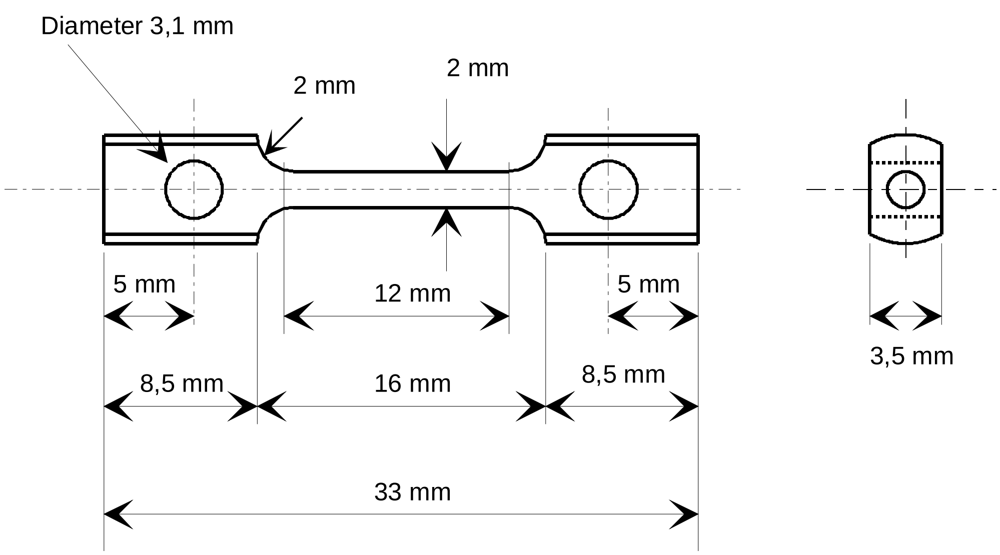

Figure 1. 2 mm diameter tensile specimens (CEA type GI-116).

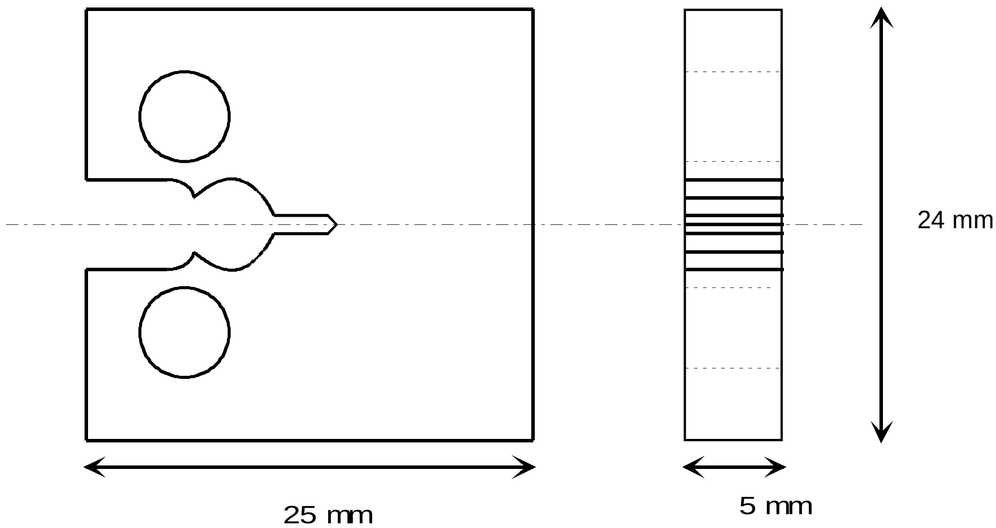

Figure 2. Sub-size CT-specimens for fracture toughness tests.

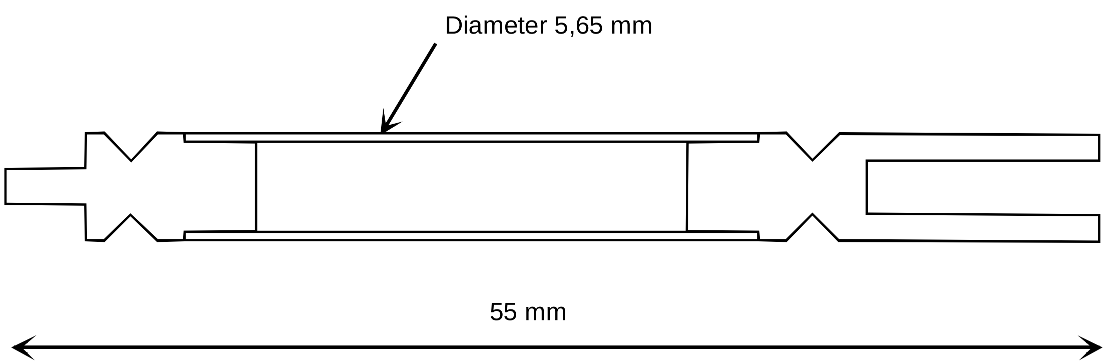

Figure 3. Pressurized tube for irradiation creep.

Chemical Composition

In preparation for the shipment from RIAR to Halden, ORNL and the University of Michigan were provided a spreadsheet containing the chemical composition of materials irradiated in the Boris 2 experiment. The same heats of 304 and 316 (i.e., 304-1 and 316-1) were used in the Boris 2 and that of the higher fluence Boris 5 and 6 irradiations. This spreadsheet is included as Appendix A of this report.

II. Status

ORNL and the University of Michigan collaborated with Faiza Sefta of EDF and Torill Karlsen and Helge Valseth of the Institute for Energy (IFE) in Norway in support of the sample shipment from RIAR (Russia) to the Halden Reactor (Norway). Preparation for the first leg of the material transport involved (1) contracting between RIAR and Halden for the transfer of samples and (2) finalization of required licensing for the shipment, which was completed in late July 2016. Halden received the samples on August 23, 2016.

Following arrival of the samples at the Halden Reactor, additional work has been initiated in preparing the samples for shipment to ORNL. This work includes:

- fabrication of an inner cask container,
- decontamination and preparation of a Type A container,
- preparation of a new activity calculation,
- all necessary internal and external paperwork, and
- handling / packaging of the samples.

A list of contact names and information associated with this effort follows:

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III. Summary

There is a collaborative effort in progress for the safe transport of nine high dpa specimens from Dimitrovgrad, Russia, to Halden, Norway, to Oak Ridge, Tennessee. Much of the effort so far has been to acquire the proper license(s) and assure the material is appropriately packaged for the first shipment between Russia and Norway. The material arrived in Halden, Norway, on August 23, 2016. Currently, new activity calculations and additional packaging and documentation efforts are being pursued. ORNL personnel will continue to work with appropriate counterparts to have the material shipped from the Halden Reactor to ORNL at the earliest possible time.

Once received, specimens will undergo receipt, inventory and processing of any return cask shipment at the ORNL Irradiated Materials Examination and Testing (IMET) hot cell. At which time, smaller shipments will be made to University of Michigan for continuous extension rate testing under LWRS primary water conditions. The University is limited on the amount of activity accepted into their facility at a given time. Therefore, shipments of material will be made between the University and ORNL. At the same time, irradiated materials from other parts of the LWRS program on Mechanisms of IASCC may be combined on shipments between the Lab and University. The high fluence samples on return to ORNL will undergo extensive characterization of both the as-tested material and will also undergo further sectioning to create samples for further microstructural analysis, in situ strain testing and additional samples that will be returned later to the University for 4-point bend testing for IASCC initiation testing under representative LWR conditions.

These materials will provide unique material for examining the IASCC initiation and crack growth phenomenon occurring at very high fluences, in which changes in the microstructure due to radiation-induced defect size, swelling and precipitation may result in different physical responses of the material than are seen at low fluence conditions. Microstructural information from these high fluence samples will provide additional data for examination of void development and swelling in high fluence materials, radiation induced solute segregation at grain boundaries (note: current models suggest saturation by 10 dpa, but may further be influenced by other high fluence phenomenon occurring) and radiation induced changes to the precipitate structures within the material.

IV. References

1. LWRS-milestone report M3LW-14OR0402023, “Post-irradiation Examination and Localized Deformation Studies on Key Specimens”, September 2015.
2. Stephenson K.J., Was G.S. “Crack initiation behavior of neutron irradiated model and commercial stainless steels in high temperature water,” *J. Nucl. Mater.*, 444 (2014) 331–341.
3. Tan L., Busby J.T. “Alloying effect of Ni and Cr on irradiated microstructural evolution of type 304 stainless steels,” *J. Nucl. Mater.*, 443 (2013) 351–358.
4. Field K.G., Yang Ying, Allen T.R., Busby J.T. “Defect sink characteristics of specific grain boundary types in 304 stainless steels under high dose neutron environments,” *Acta Materialia*, 89 (2015) 438–449.

APPENDIX A
Chemical Composition of Materials Irradiated in the Boris 2 Experiment

TABLE 2.III : Materials irradiated in the Boris 2 experiment : chemical composition.

Material	Mark	Origine	Chemical composition (weight)																
			C (%)	S (%)	P (%)	Si (%)	Mn (%)	Ni (%)	Cr (%)	Mo (%)	Cu (%)	Co (%)	Nb-Ta (%)	B (ppm)	O (ppm)	N (ppm)	Al (%)	Ti (%)	Other (%)
Welded materials																			
308-1	SA	Framatome (weld)	0.006	0.012	0.018	0.38	1.84	9.65	20.30	0.16	-	0.05	<0.005	-	-	479	-	-	
308-2	SB	ESAB (wire)	0.012	0.016	0.015	0.39	1.81	9.70	20.15	0.046	-	0.049	-	-	-	-	-	-	-
Type 316 and modified 316 materials																			
316-1	B	EDF	0.054	0.022	0.027	0.68	1.12	10.60	16.60	2.25	0.24	0.12	0.01	5	41	230	-	<0.01	-
316-2	DB	Ugine (heat)	0.049	0.010	0.022	0.76	1.16	10.45	16.56	2.36	0.13	0.19	-	-	-	-	-	-	
316Fe	TB	-	0.014	-	-	0.50	-	10.53	12.99	-	-	-	-	-	-	-	-	-	-
316SG	SG	EDF (identical to 316-1-CW)	0.054	0.022	0.027	0.68	1.12	10.60	16.60	2.25	0.24	0.12	0.01	5	41	230	-	<0.01	-
316LG	LG	EDF (identical to 316-1-CW)	0.054	0.022	0.027	0.68	1.12	10.60	16.60	2.25	0.24	0.12	0.01	5	41	230	-	<0.01	-
316Ti73-CW	E	-	0.049	0.01	<0.005	0.33	1.48	13.0	16.9	2.67	0.04	-	-	<10	28	320	-	0.25	-
316Ti73-CWA	F	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
316Zr	BZ	-	0.046	-	-	0.72	-	-	-	-	-	-	-	-	-	-	-	-	Zr : 0.38
316Hf	BH	-	0.049	-	-	0.72	-	-	-	-	-	-	-	-	-	-	-	-	Hf : 0.38
316HP	PB	ENSME	0.01	0.001	<0.001	-	1.3	13.6	17.5	2.4	-	-	-	-	7	10	-	-	-
316HPSi	PC	ENSME	0.05	0.002	<0.001	0.8	1.3	13.8	17.5	2.4	-	-	-	-	4	10	-	-	-
316Mono	MC	ENSME	-	-	-	-	-	14.5	15.2-16.7	1.8-2.4	-	-	-	-	-	-	–	-	-
Type 304 and modified 304 materials																			
304-1	A	Creusot-Loire (heat)	0.022	0.0007	0.032	0.36	1.79	9.86	18.61	-	0.25	0.064	-	9	-	610	-	-	-
304-2	CB	Creusot-Loire (heat)	0.025	0.001	0.026	0.49	1.70	9.11	19.12	-	0.166	0.056	-	7	-	680	-	-	-
304Fe	VB	-	-	-	-	-	-	9.14	12.97	-	-	-	-	-	-	-	-	-	-
304Zr	AZ	-	0.023	-	-	0.40	-	-	-	-	-	-	-	-	-	-	-	-	Zr : 0.38
304Hf	AH	-	0.017	-	-	0.04	-	-	-	-	-	-	-	-	-	-	-	-	Hf : 0.38
321PX	RB	-	0.059	0.013	0.027	0.51	1.67	10.10	17.90	0.24	-	-	<0.05	18	-	9	-	0.59	-
Other materials																			-
Uranus S1N	U	-	0.009	0.008	0.010	4.09	1.64	14.48	17.52	0.035	0.03	-	0.086	11	-	260	0.14	-	-
N9	NB	-	0.068	0.002	0.008	0.76	1.82	25.74	11.68	1.74	-	-	-	65	-	45	0.009	0.51	Zr : 0.0011
HNi-Mono	MN	-	0.11	-	-	-	-	bal.	16	-	-	-	3.5	-	-	-	3.5	3.5	-

***Note: 304H means SA304 (the H is for SA in French) and 316E means CW316 (écroui = CW in French). Heats 304-1 and 316-1 are reference heats for EDF; they were kept out of the CIR program.**