

# NSUF Tensile Testing and Characterization of Irradiated Grade 92 Ferritic-Martensitic Steels



Ben Garrison  
Weicheng Zhong  
Lizhen Tan  
Kory Linton

**May 2019**

**Approved for Public Release.  
Distribution is Unlimited.**

#### DOCUMENT AVAILABILITY

Reports produced after January 1, 1996, are generally available free via US Department of Energy (DOE) SciTech Connect.

**Website** [www.osti.gov](http://www.osti.gov)

Reports produced before January 1, 1996, may be purchased by members of the public from the following source:

National Technical Information Service  
5285 Port Royal Road  
Springfield, VA 22161  
**Telephone** 703-605-6000 (1-800-553-6847)  
**TDD** 703-487-4639  
**Fax** 703-605-6900  
**E-mail** [info@ntis.gov](mailto:info@ntis.gov)  
**Website** <http://classic.ntis.gov/>

Reports are available to DOE employees, DOE contractors, Energy Technology Data Exchange representatives, and International Nuclear Information System representatives from the following source:

Office of Scientific and Technical Information  
PO Box 62  
Oak Ridge, TN 37831  
**Telephone** 865-576-8401  
**Fax** 865-576-5728  
**E-mail** [reports@osti.gov](mailto:reports@osti.gov)  
**Website** <http://www.osti.gov/contact.html>

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

Reactor and Nuclear Sciences Division

**NSUF TENSILE TESTING AND CHARACTERIZATION OF IRRADIATED GRADE 92  
FERRITIC-MARTENSITIC STEELS**

Ben Garrison  
Weicheng Zhong  
Lizhen Tan  
Kory Linton

May 2019

Prepared by  
OAK RIDGE NATIONAL LABORATORY  
Oak Ridge, TN 37831-6283  
managed by  
UT-BATTELLE, LLC  
for the  
US DEPARTMENT OF ENERGY  
under contract DE-AC05-00OR22725



## CONTENTS

FIGURES .....	v
TABLES .....	v
ACRONYMS .....	vii
ACKNOWLEDGMENTS .....	ix
EXECUTIVE SUMMARY .....	xi
1. INTRODUCTION .....	1
2. TENSILE TESTING EXPERIMENTAL PROCEDURE.....	1
3. MATERIAL CHARACTERIZATION.....	2
4. RESULTS .....	5
5. CONCLUSIONS AND FUTURE WORK .....	8
6. REFERENCES .....	8



## FIGURES

Figure 1.	Instron ElectroPuls E1000 mechanical testing system at Irradiated Materials Examination and Testing facility. ....	2
Figure 2.	Schematic of one piece of an SS-J2 specimen after tensile testing to fracture showing caliper measurement locations. ....	3
Figure 3.	Fracture surfaces for specimens of Grade 92 (GB03 and GB10), 800H (AR2), and 800H-TMP (HG1). ....	4
Figure 4.	Particles inside dimples: (a) niobium-containing particles in GB03 and (b) titanium-containing particles in AR2. ....	4
Figure 5.	Tension test results for (a) TC03, (b) TN03, (c) VN03, (d) KGT 1791, (e) KGT 1735, and (f) KGT 1783. ....	6
Figure 6.	Tension test results for (a) KGT 2596, (b) KGT 2578, (c) KGT 2597, (d) KGT 1772, (e) KGT 1712, and (f) KGT 1806. ....	7

## TABLES

Table 1.	Oak Ridge National Laboratory High Flux Isotope Reactor–Irradiated Tensile Test Specimens .....	1
Table 2.	Idaho National Laboratory Advanced Test Reactor–Irradiated Tensile Test Specimens .....	2





## ACRONYMS

ATR	Advanced Test Reactor (INL)
EBSD	electron backscattered diffraction
EDS	energy dispersive x-ray spectroscopy
FM	ferritic-martensitic (steel)
FIB	focused-ion beam
HFIR	High Flux Isotope Reactor
IMET	Irradiated Materials Examination and Testing (hot cell facility)
INL	Idaho National Laboratory
LANL	Los Alamos National Laboratory
LAMDA	Low Activation Materials Development and Analysis (laboratory)
LWR	light water reactor
NSUF	Nuclear Science User Facility
ORNL	Oak Ridge National Laboratory
PIE	postirradiation examination
TEM	transmission electron microscopy



## **ACKNOWLEDGMENTS**

This research used resources at the Low Activation Materials Development and Analysis laboratory operated by the Oak Ridge National Laboratory. Funding for this research was provided by the US Department of Energy, Office of Nuclear Energy Nuclear Science User Facility. This report was authored by UT-Battelle, LLC under Contract DEAC05-00OR22725 with the US Department of Energy.



## **EXECUTIVE SUMMARY**

This report summarizes the completed post-irradiation tensile testing of five ferritic-martensitic steel SS-J2 specimens at the Irradiated Materials Examination and Testing hot cell facility at Oak Ridge National Laboratory. A brief description of the motivation for the study, the properties and irradiation conditions of the samples, and the tensile testing conditions are provided. The results will be used to compare the mechanical properties of the irradiated specimens to unirradiated conditions to determine how irradiation affects the steel alloy material behavior.

## 1. INTRODUCTION

To extend the life of existing light water reactors (LWRs), understanding correlations between mechanical and microstructural properties of their structural components is required, especially with radiation effects on these components from reactor use. To this end, ferritic-martensitic (FM) steel Grade 92 (9Cr-1.8W-0.5MoVNb) and austenitic alloy 800H (20Cr-32Ni-TiAl) were selected for examination of neutron radiation effects on their mechanical and microstructural properties. Not only will these results be useful in evaluating the safety of structural components in current reactors, but they will also provide insights for the development of new advanced alloys, both of which will benefit advanced reactor technologies, small modular reactors, and LWR sustainability programs.

This is the fifth in a series of progress reports on this project. The first one (M3UF-18OR0210032) summarized tensile testing of High Flux Isotope Reactor (HFIR)-irradiated Grade 92 specimens at the Irradiated Materials Examination and Testing (IMET) hot-cell facility [1]. The second one (M4NA-17OR0204015) summarized identification and shipment progress for the Idaho National Laboratory (INL) and Los Alamos National Laboratory (LANL) samples sent for examination at Oak Ridge National Laboratory (ORNL) [2]. The third one (M3NA-17OR0204016) updated the postirradiation examination (PIE) progress on the ORNL samples, including tensile and hardness tests and microstructural characterization [3]. The fourth one (M2NA-17OR0204014) summarized the first year's progress on this project, including sample procurement and PIE status [4]. This fifth report will summarize tensile test results and microstructural characterization for irradiated samples from INL and ORNL.

The Nuclear Science User Facility (NSUF) at ORNL has numerous PIE capabilities including in-cell tensile testing of irradiated subsized (SS-J2) specimens at IMET and microstructural characterization of irradiated specimens at the Low Activation Materials Development and Analysis (LAMDA) laboratory. Included in this report is a summary of tensile test results for the last set of specimens, including six irradiated FM steel specimens and six irradiated austenitic alloy specimens, which were irradiated at ORNL's HFIR and INL's Advanced Test Reactor (ATR). Also included are representative fractography measurements of previously tensile-tested irradiated Grade 92 and 800H specimens, which have been performed at LAMDA. More detailed PIE work will be performed in the near future, including fractographic analysis; Vickers hardness and nanoindentation testing; and focused-ion beam (FIB), transmission electron microscopy (TEM), electron backscattered diffraction (EBSD), and energy dispersive x-ray spectroscopy (EDS) imaging.

## 2. TENSILE TESTING EXPERIMENTAL PROCEDURE

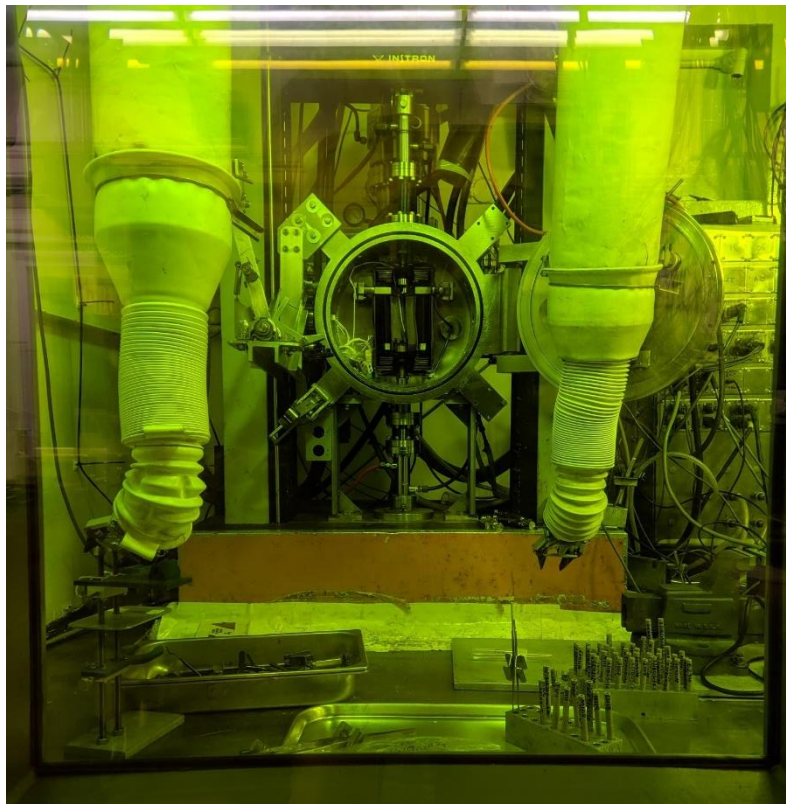
Three HFIR-irradiated FM SS-J2 specimens and nine ATR-irradiated austenitic and FM SS-J2 specimens were tensile tested with an Instron ElectroPuls E1000 test system located in Cell 2 in the IMET facility (shown in Figure 1). Tables 1 and 2 show the specimens tested with their respective irradiation conditions. The SS-J2 specimens had dimensions of  $16 \times 4 \times 0.5$  mm with a gauge section of  $5 \times 1.2 \times 0.5$  mm. The crosshead speed during testing was kept constant at 0.012 in./min (0.00508 mm/s). The load versus crosshead displacement response of each specimen was measured throughout the test so the stress strain curves could be calculated. Tensile tests were performed through specimen fracture.

**Table 1. Oak Ridge National Laboratory High Flux Isotope Reactor-Irradiated Tensile Test Specimens**

Sample ID	Model Alloy	Dose [dpa]	Target Temperature [°C]
TC03	TaC-engineered	7.44	300
TN03	TaN-engineered	7.44	300
VN03	VN-engineered	7.44	300

**Table 2. Idaho National Laboratory Advanced Test Reactor–Irradiated Tensile Test Specimens**

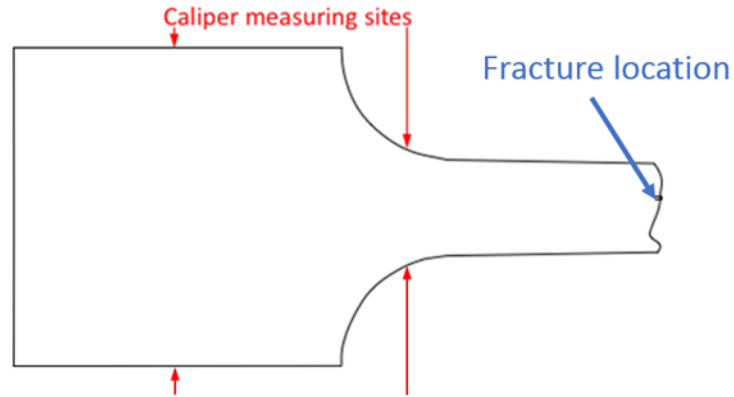
<b>Sample ID</b>	<b>Material Alloy</b>	<b>Dose [dpa]</b>	<b>Temperature [°C]</b>
KGT 1772	800H	3.9	451.5
KGT 1712	800H	7.27	359.0
KGT 1806	800H	9.01	431.0
KGT 2596	800H-TMP	3.95	451.5
KGT 2578	800H-TMP	7.36	359.0
KGT 2597	800H-TMP	9.12	431.0
KGT 1791	NF616	2.96	291.5
KGT 1735	NF616	5.91	359.0
KGT 1783	NF616	8.16	431.0



**Figure 1. Instron ElectroPuls E1000 mechanical testing system at Irradiated Materials Examination and Testing facility.**

### **3. MATERIAL CHARACTERIZATION**

Currently, all specimens listed in Tables 1 and 2 are located in LAMDA, where experimental specimen thickness measurements will be taken at the end-tab section and near the shoulder of each specimen using calipers. The measurement locations are shown in Figure 2. The measurements will be used to justify or correct the stress of the stress-strain curves. When measurements are complete, fractography, polishing, Vickers hardness, nanoindentation, FIB, TEM, EBSD, and EDS imaging will be completed.

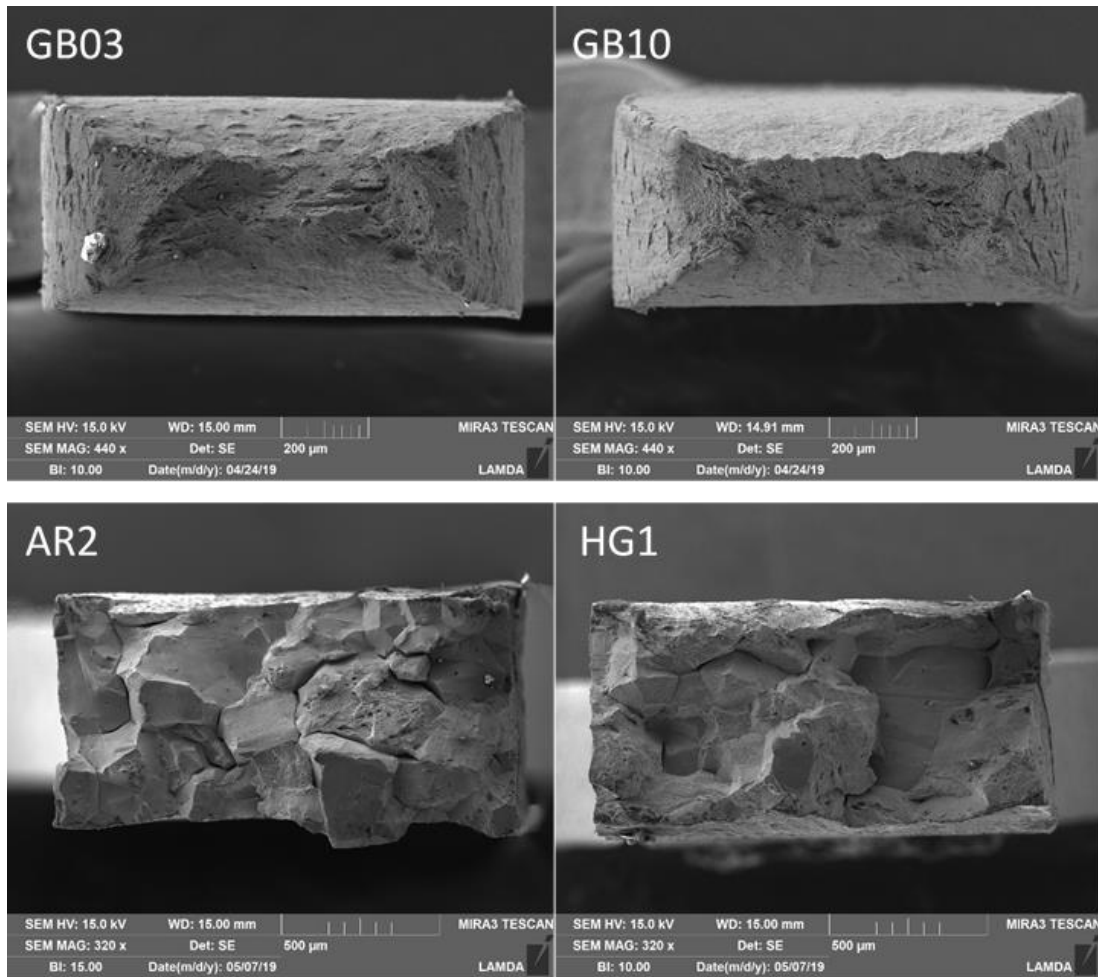


**Figure 2. Schematic of one piece of an SS-J2 specimen after tensile testing to fracture showing caliper measurement locations.**

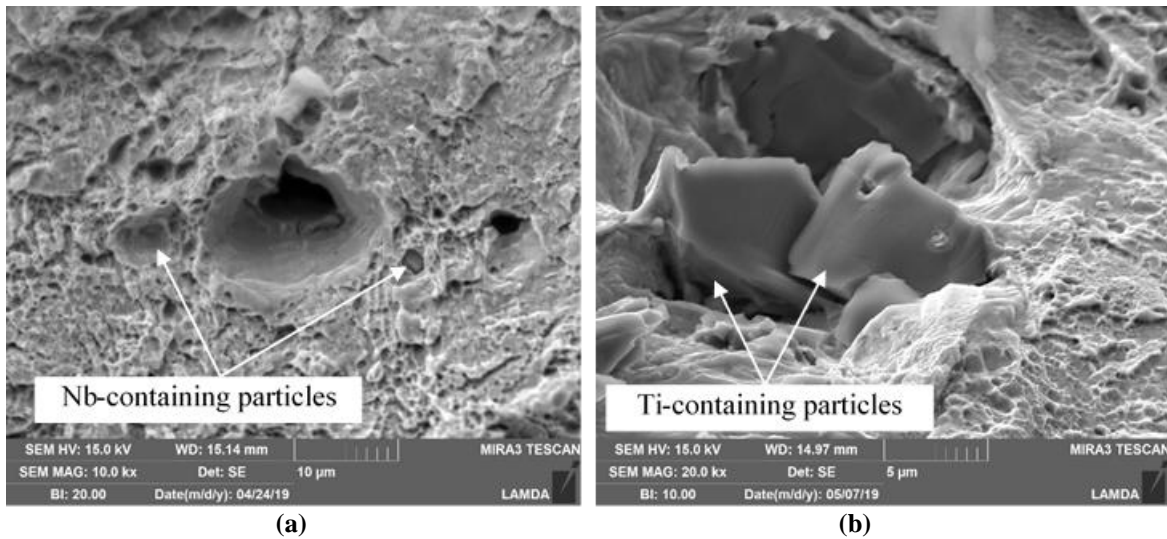
Representative fractography results for specimens tensile-tested for the previous milestone report (M2NA-17OR0204014) are shown in Figure 3: Grade 92 specimens GB03 (0.52 dpa at nominal 300°C) and GB10 (0.46 dpa at nominal 650°C), alloy 800H specimen AR2 (1.28 dpa at 580°C), and alloy 800H-TMP specimen HG1 (1.28 dpa at 580°C). Samples GB03 and GB10 show similar ductile fracture surfaces, and significant necking was exhibited. Large (5–10  $\mu\text{m}$ ) and small (submicron) dimples were observed on the fracture surfaces of both GB03 and GB10. Figure 4a shows representative dimples of GB03. Particles observed inside some dimples were identified as niobium-containing particles from their EDS measurements.

The 800H (AR2) and 800H-TMP (HG1) specimens were tensile-tested at 580°C. Both AR2 and HG1 demonstrate primarily brittle fracture surfaces with negligible necking. Both samples have large intergranular fractured grains with some cleavage features and large dimples. Large particles (over 5  $\mu\text{m}$ ) were observed inside the dimples. Figure 4b shows the particles in the AR2 sample, which were identified as titanium-containing particles.





**Figure 3. Fracture surfaces for specimens of Grade 92 (GB03 and GB10), 800H (AR2), and 800H-TMP (HG1).**



**Figure 4. Particles inside dimples: (a) niobium-containing particles in GB03 and (b) titanium-containing particles in AR2.**

#### **4. RESULTS**

The tensile stress-strain curves calculated from the load vs. crosshead displacement data are shown in Figure 5 for FM steels (TC03, TN03, VN03, KGT 1791, KGT 1735, and KGT 1783) and Figure 6 for austenitic alloys (KGT 1772, KGT 1712, KGT 1806, KGT 2596, KGT 2578, and KGT 2597). These curves will be used to calculate the yield strength, ultimate strength, uniform elongation, and total elongation. These mechanical properties will be compared with data in the literature for similar alloys and will be used to evaluate changes in mechanical performance with irradiation temperature and dose.

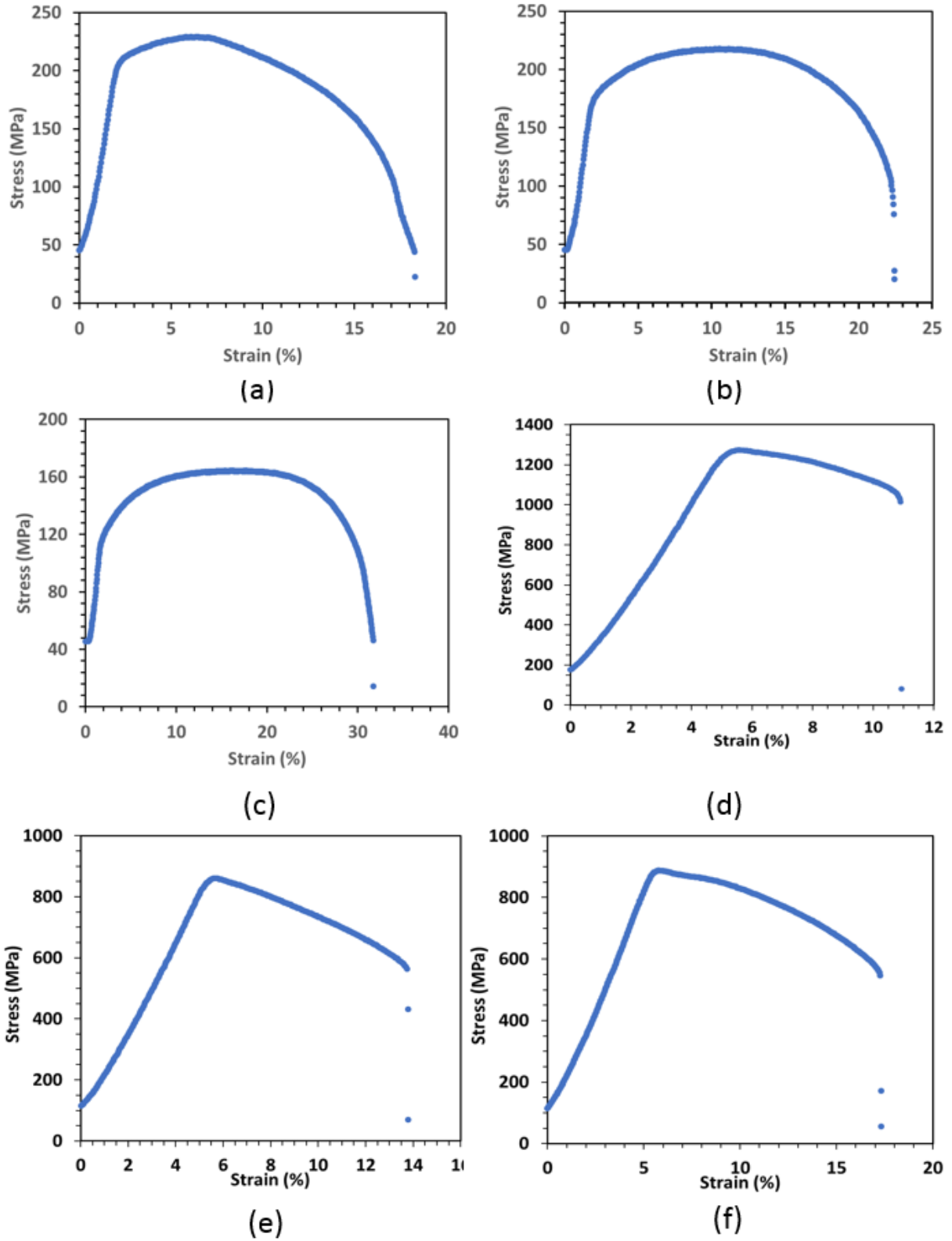
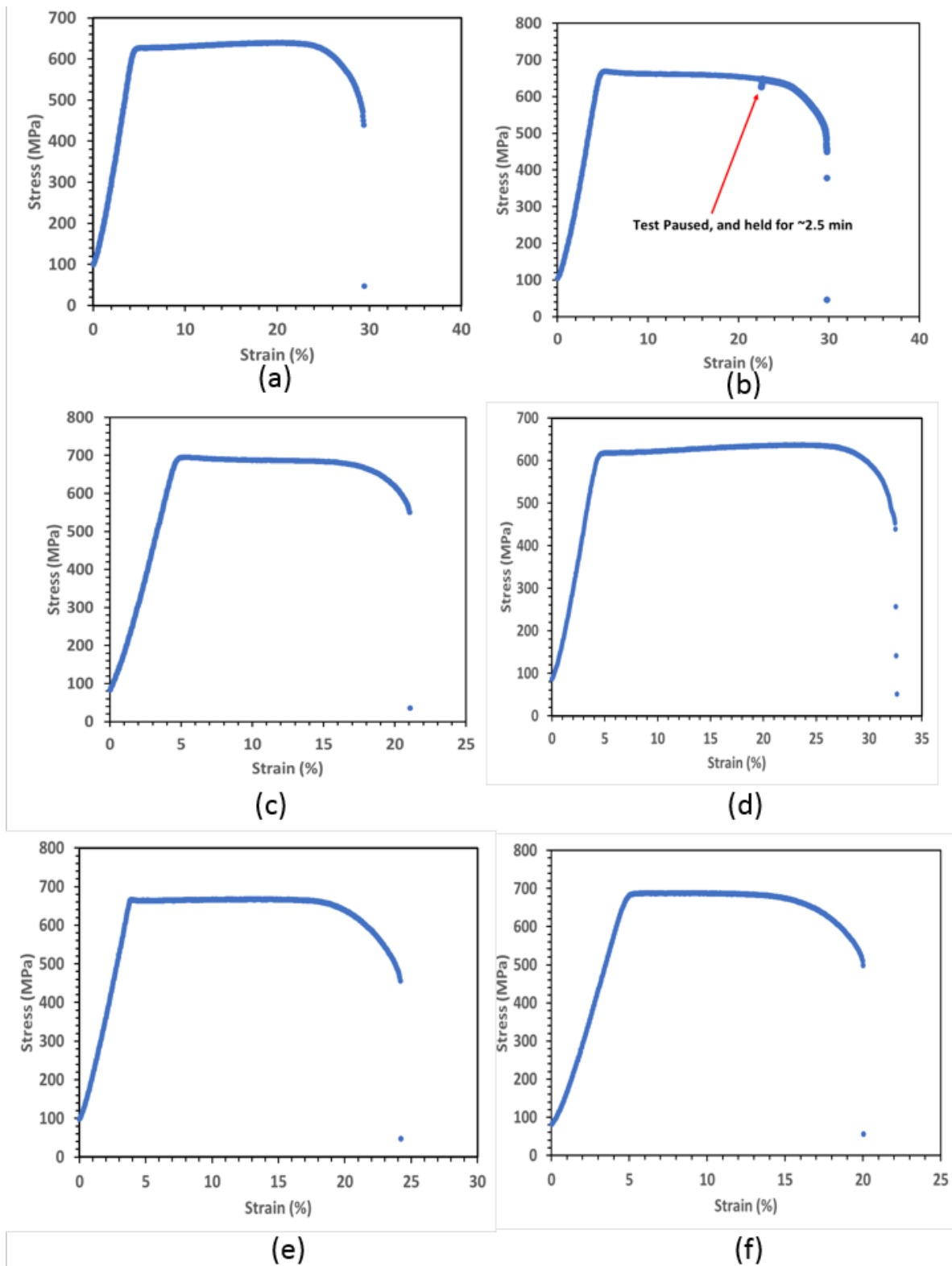


Figure 5. Tension test results for ferritic-martensitic steel specimens (a) TC03, (b) TN03, (c) VN03, (d) KGT 1791, (e) KGT 1735, and (f) KGT 1783. More information can be found in tables 1 and 2.



**Figure 6. Tension test results for austenitic alloy 800H specimens (a) KGT 2596, (b) KGT 2578, (c) KGT 2597, KGT 1772, (e) KGT 1712, and (f) KGT 1806. As shown in (b), testing for KGT 2578 was paused and held for 2.5 minutes before continuing tensile extension; however, it was still possible to obtain mechanical properties such as yield stress and ultimate strength. More material information can be found in table 2.**

## 5. CONCLUSIONS AND FUTURE WORK

The 2019 NSUF Milestone, M3UF-19OR0210022, titled “Report on In-Cell Tensile Testing of Specimens Received from LANL and INL” was completed with tensile tests of three FM HFIR-irradiated specimens and nine austenitic and FM ATR-irradiated specimens at the IMET facility followed by specimen transfers to the LAMDA facility and cross-section measurements. Fractography analysis was completed on two HFIR-irradiated FM steel specimens and two HFIR-irradiated austenitic alloy specimens. Future work for this research involves hardness measurements using Vickers and nanoindentation and microstructure characterization of the specimens. Microstructure characterization will include fractography, FIB, TEM, EBSD, and EDS. The combination of these mechanical property measurements with future microstructure characterizations will produce a thorough evaluation of FM and austenitic steel performance as a structural nuclear component after exposure to reactor conditions and will provide an in-depth understanding of microstructure evolution for future alloy development.

## 6. REFERENCES

1. A. Raftery, L. Tan, H. Sakasegawa, K. Linton, *Tensile Testing of Irradiated Grade 92 Ferritic-Martensitic Steels at the IMET Hot Cell Facility*, ORNL/LTR-2018/499, April 2018.
2. L. Tan, K. Linton, C. Knight, T. Saleh, *Identification and Shipment Progress for the INL and LANL Samples to Be Received at ORNL*, ORNL/LTR-2019/1083, February 2019.
3. L. Tan, T. Chen, Partial Completion of Post-Irradiation Examination of the ORNL Samples, ORNL/TM-2019/1135, February 2019.
4. L. Tan, T. Chen, K. Linton, C. Knight, T. Saleh, *First Annual Progress Report on the Procurement and Post-Irradiation Examination of the Selected Samples of Alloy 800H and Grade 92 and 91 Steels*, ORNL/TM-2019/1136, February 2019.