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Disassembly of Capsules after the Irradiation of Prototype Metal and Nanocomposite Specimens in the High Flux Isotope Reactor

Jesse Werden
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OAK RIDGE NATIONAL LABORATORY
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Nuclear Energy and Fuel Cycle Division

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ACRONYMS

DOE	US Department of Energy
GENTEN	general tensile
HFIR	High Flux Isotope Reactor
HT	hydraulic tube
IMET	Irradiated Materials Examination and Testing Facility
LAMDA	Low Activation Materials Development and Analysis Laboratory
MIT	Massachusetts Institute of Technology
ORNL	Oak Ridge National Laboratory
SiC	silicon carbide
TM	thermometry
TRRH	target rod rabbit holder

SUMMARY

This report summarizes the disassembly, thermometry (TM) analysis, and future post-irradiation examination of irradiation capsules that contain nanodispersion-strengthened materials for the improved neutron irradiation resistance of fuel cladding and reactor core materials. All six capsules were successfully disassembled, and all TM was shipped to the Low Activation Materials Development and Analysis Laboratory for further analysis. The results of this project will support the development of new radiation-resistant materials by helping researchers understand the mechanism of defect evolution at interfaces in nanodispersion-strengthened materials.

1. INTRODUCTION

The Massachusetts Institute of Technology (MIT) is currently studying different fuel cladding and reactor core materials with improved neutron irradiation resistance due to nanodispersions at 0, 1, or 2 dimensions; these values correspond to particles, nanotubes, and sheets, respectively. The purpose of this project is to perform neutron irradiation tests on several nanodispersion-strengthened materials to provide data on defect mechanisms at the nanoscale.

Thirteen nanodispersion-strengthened materials were irradiated in Oak Ridge National Laboratory's (ORNL) High Flux Isotope Reactor (HFIR). Six irradiation capsules were assembled and irradiated at a target temperature of 300 ± 50°C with approximate doses of 0.7, 1.4, and 2.1 dpa (two capsules per irradiation condition). This report presents the disassembly of these capsules, thermometry (TM) analysis, and future post-irradiation examination.

1.1 CAPSULE DESIGN

The general tensile (GENTEN) irradiation capsule design comprises three specimen holders stacked axially within the rabbit housing, as shown in Figure 1. Each holder contains 12 SSJ2 tensile specimens and four passive silicon carbide (SiC) TMs. Therefore, each capsule contains 36 tensile specimens and 12 SiC TMs. Chevrons are used as filler pieces to produce a uniform thermal load, and spring pins secure all specimens in place. The holders feature centering tabs to keep them centered inside the housing and thus maintain a constant gas gap between the holder and housing. Compression springs are placed on both ends of the internal assembly to minimize axial heat loss. Six capsules, labeled as JULI01 through JULI06, were assembled. Figure 2 shows an example of the parts layout for one capsule before assembly.

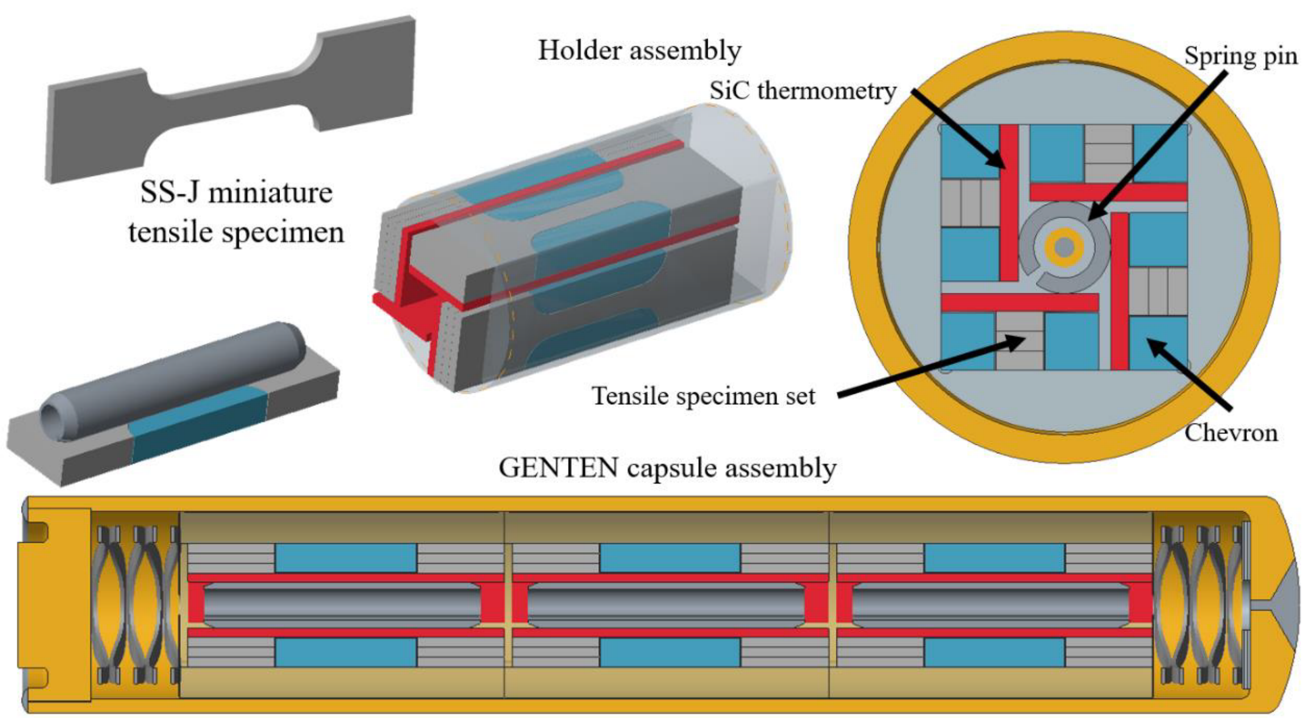

Figure 1. Irradiation capsule design for tensile specimens [1].

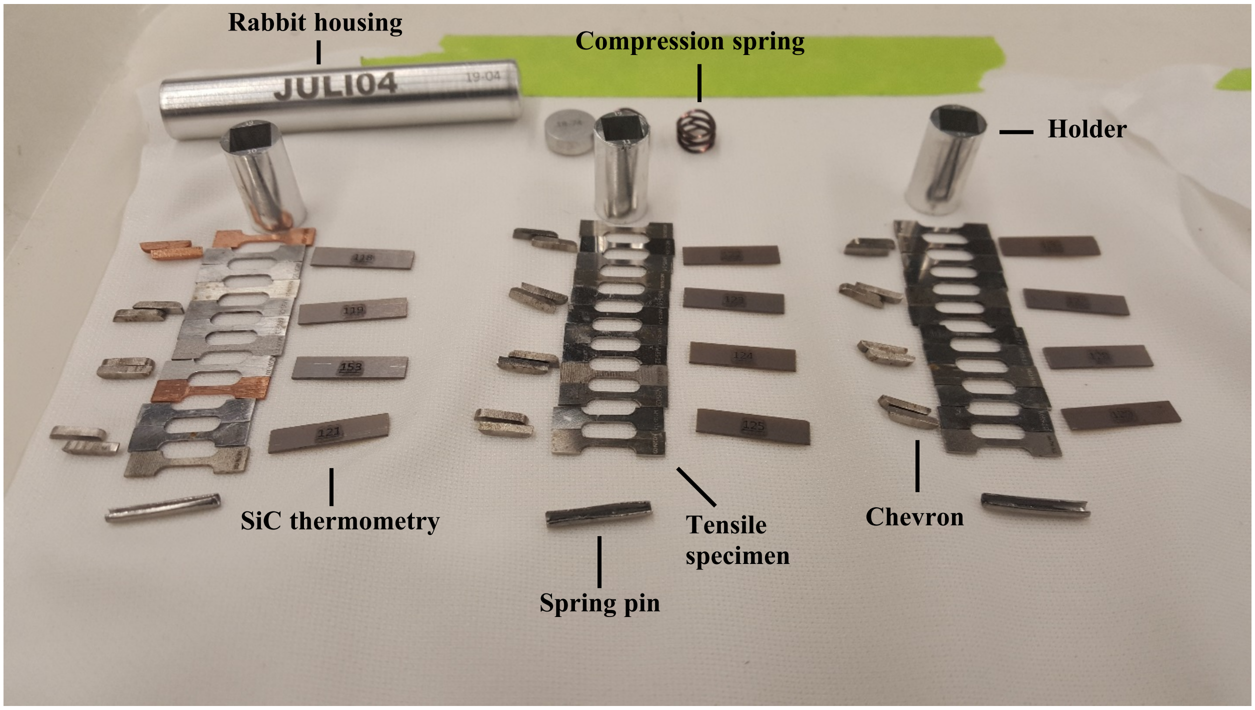

Figure 2. Parts layout for GENTEN irradiation capsule JULI04 [1].

1.2 TEST MATRIX

Table 1 summarizes the irradiation test matrix for this project. These six irradiation capsules contain 15 different materials provided by MIT and ORNL. All irradiation design temperatures are 300 ± 50°C with doses of approximately 0.7, 1.4, and 2.1 dpa, and these values correspond to approximately one-half, one, and two HFIR cycles, respectively.

Table 1. Irradiation test matrix [1].

Capsule ID	JULI01	JULI02	JULI03	JULI04	JULI05	JULI06	
Dose (dpa)	0.7		1.4		2.1		
Materials	Number of SSJ2 specimens						Total
Al	3	2	3	2	3	2	15
Al + CNT	3	2	3	2	3	2	15
Cu	3	1	3	1	3	1	12
Cu + graphene	3	1	3	1	3	1	12
Fe-16Cr-2Si	2	3	2	3	2	3	15
Fe-20Cr-2Si	2	3	2	3	2	3	15
Grade 91	3	3	3	3	3	3	18
Ni	2	3	2	3	2	3	15
Ni + CNT	3	2	3	2	3	2	15
OFRAC	0	3	0	3	0	3	9
Single crystal Ni	2	3	2	3	2	3	15
Steel 1	3	2	3	2	3	2	15
Steel 1 + oxide/carbide	2	3	2	3	2	3	15
Steel 2	2	3	2	3	2	3	15
Steel 2 + oxide/carbide	3	2	3	2	3	2	15
Total number of specimens	36	36	36	36	36	36	216

2. IRRADIATION HISTORY

The six JULI capsules were irradiated in HFIR during cycle 485a (December 17–26, 2019), 485b (January 3–19, 2020), and 486 (January 30–February 24, 2020). The hydraulic tube (HT) irradiation facility was used for capsules with an irradiation time of less than one cycle. A target rod rabbit holder (TRRH) irradiation facility allowed the capsule irradiation for one or two full HFIR cycles. One HFIR cycle is approximately 25 days. Table 2 shows the irradiation details for each capsule.

Table 2. Irradiation history of the JULI capsules.

Capsule ID	Beginning cycle	End cycle	Irradiation facility	Location	Axial position	Time in the reactor
JULI01	485a	485b	HT	B3	3	12.5 days
JULI02	485a	485b	HT	B3	7	12.5 days
JULI03	485a	485b	TRRH	G5	6	1 cycle
JULI04	485a	485b	TRRH	E7	6	1 cycle
JULI05	485a	486	TRRH	B1	6	2 cycles
JULI06	485a	486	TRRH	C1	6	2 cycles

3. IRRADIATION CAPSULE DISASSEMBLY

The six GENTEN capsules (JULI01–JULI06) were successfully disassembled in hot cell #6 of ORNL's Irradiated Materials Examination and Testing Facility (IMET). The first step in disassembling the capsules was to cut both ends of the capsule housing by using a double-bladed low-speed saw in which the distance between the blades is set to not harm any of the specimens. The capsule was then moved to a steel tray onto which the contents could be safely extracted. Figure 3 shows the JULI06 capsule as an example in which both sides of the housing were cut and the three holders were extracted. Next, a dental pick was used to push out the spring pin from the center of each holder. Once the spring pin was removed, the tensile specimens and SiC TMs fell out of the holder. Figure 4 shows the parts layout for the completely disassembled JULI01 capsule.

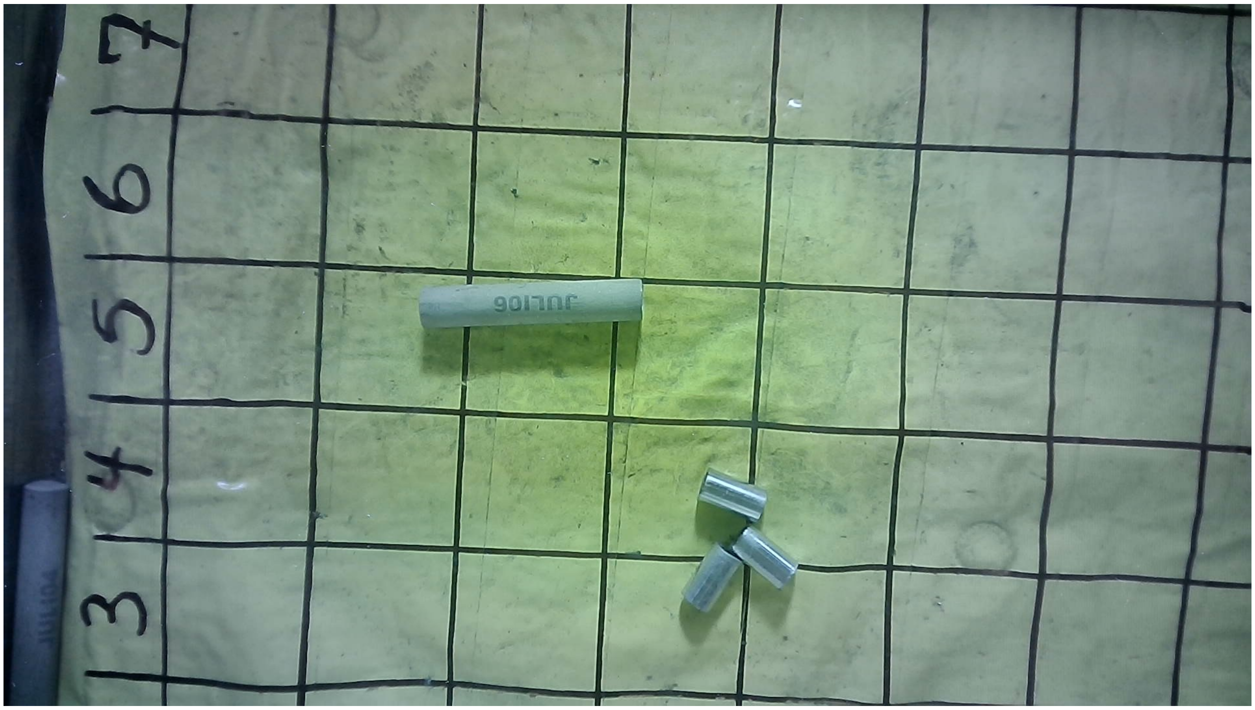

Figure 3. Three holders removed from the JULI06 capsule.

Figure 4. Disassembled JULI01 capsule.

Each tensile specimen was then sorted and placed into individual fiber tubes marked with its specimen ID. Several SiC TMs were broken during disassembly; however, each capsule had enough intact TMs for post-irradiation analysis. All SSJ2 tensile specimens were recovered intact, and images of individual specimens are shown in Appendix A. All TM specimens were shipped to the Low Activation Materials Development and Analysis Laboratory (LAMDA) for dilatometry analysis, and all tensile specimens remained at IMET for tensile testing in hot cell #1.

4. THERMOMETRY

All TMs in each capsule were recovered during disassembly. Table 3 shows the status of the TMs recovered from each capsule. Three TMs per capsule were selected to be analyzed via dilatometry [2] to confirm the irradiation temperature; the results are shown in Table 4. GENTEN design calculations in Piela et al. [1], Le Coq et al. [3], and Howard and Smith [4] show that the TM temperature is on average 15°C higher than the specimens temperature. Thus, the experimental specimen temperature was estimated to be 15°C lower than the average temperature of the TMs for each capsule. The specimen temperatures obtained by this approach are in agreement with the target irradiation temperature (300 ± 50°C).

Table 3. Status of all TMs recovered during the disassembly of JULI capsules.

Capsule ID	Holder	TM ID	Status	Capsule ID	Holder	TM ID	Status
JULI01	Top holder	081	Intact	JULI04	Top holder	118	Intact
		082	Intact			119	Intact
		083	Intact			153	Intact
		084	Intact			121	Intact
	Middle holder	085	Intact		Middle holder	122	Intact
		086	Intact			123	Intact
		087	Intact			124	Intact
		088	Intact			125	Intact
	Bottom holder	089	Intact		Bottom holder	126	Intact
		090	Intact			127	Intact
		091	Intact			128	Intact
		092	Intact			129	Intact
JULI02	Top holder	093	Broken in half	JULI05	Top holder	130	Intact
		094	Intact			131	Intact
		095	Intact			132	Intact
		096	Intact			133	Intact
	Middle holder	097	Intact		Middle holder	105	Intact
		098	Intact			135	Intact
		099	Intact			136	Intact
		100	Intact			137	Intact
	Bottom holder	101	Intact		Bottom holder	138	Two chipped corners
		102	Intact			139	Intact
		103	Intact			140	Intact
		104	Intact			141	Intact
JULI03	Top holder	106	Intact	JULI06	Top holder	142	Broken in half
		107	Intact			143	Intact
		108	Intact			144	Intact
		109	Intact			145	Intact
	Middle holder	110	Broken		Middle holder	146	Intact
		111	Chipped edge			147	Intact
		112	Intact			148	Intact
		113	Intact			149	Intact
	Bottom holder	114	Broken		Bottom holder	150	Broken
		115	Intact			151	Intact
		116	Intact			152	Intact
		117	Intact			154	Intact

Table 4. TM dilatometry results and estimated temperatures of the tensile specimens.

Capsule ID	TM ID	TM temperature (°C)	TM average temperature (°C) ± σ	Specimen temperature (°C)
JULI01	081	304	367 ± 44	352
	085	398		
	089	399		
JULI02	094	297	300 ± 5	285
	097	307		
	101	318		
JULI03	106	282	295 ± 16	280
	107	284		
	113	291		
JULI04	118	269	306 ± 27	291
	122	322		
	126	329		
JULI05	130	331	347 ± 24	332
	105	331		
	138	381		
JULI06	143	327	328 ±12	313
	146	343		
	151	314		

5. FUTURE WORK

MIT specimen tensile testing will be performed in IMET cell #1. Two or three specimens per material per irradiation condition will be tested. Additionally, unirradiated specimens of the same materials will be tested. The MIT tensile test matrix will follow ASTM E8a with a strain rate of 0.018 mm/min to determine material yield strengths. Table 5 lists the specimens that will be tensile tested.

Table 5. Specimens to be tensile tested.

Dpa	Capsule ID	Material	Specimen ID
0	N/A	Al	M1A 17
		Al	M1A 18
		Al	M1A 19
		Al + CNT	M2A 04
		Al + CNT	M2A 06
		Al + CNT	M2A 14
		Fe-16Cr-2Si	M3S 16
		Fe-16Cr-2Si	M3S 17
		Fe-16Cr-2Si	M3S 18
		Fe-20Cr-2Si	M4S 16
		Fe-20Cr-2Si	M4S 17
		Fe-20Cr-2Si	M4S 18
		Cu	M5C 13
		Cu	M5C 14
		Cu	M5C 15
		Cu + CNT	M6C 06
		Cu + CNT	M6C 11
		Cu + CNT	M6C 15
		Single crystal Ni	M7N 16
		Steel 1	M8S 08
		Steel 1	M8S 09
		Steel 1	M8S 11
		Steel 2	M9S01
		Steel 2	M9S02
		Steel 2	M9S03
		Steel 1 + OC	M10S 17
		Steel 1 + OC	M10S 18
		Steel 1 + OC	M10S 19
		Steel 2 + OC	M11S 01
		Steel 2 + OC	M11S 02
		Steel 2 + OC	M11S 03
		Ni	M12N 11
		Ni	M12N 16
		Ni	M12N 19
0.7	JULI01	Ni + CNT	M13N 14
		Ni + CNT	M13N 16
		Ni + CNT	M13N 17
		Cu	M5C 02
		Al	M1A 01
		Al	M1A 02

		Cu + CNT	M6C 04
		Al + CNT	M2A 09
		Cu + CNT	M6C 05
		Al	M1A 03
		Ni + CNT	M13N 10
		Steel 1	M8S 01
		Steel 2	M9S08
		Ni + CNT	M13N 11
		Steel 1 + OC	M10S 01
		Steel 2 + OC	M11S 08
		Single crystal Ni	M7N 01
		Fe-16Cr-2Si	M3S 01
		Fe-20Cr-2Si	M4S 06
		Ni	M12N 01
		Steel 2 + OC	M11S 09
		Steel 1	M8S 02
		Ni + CNT	M13N 12
		Steel 2	M9S09
		Steel 1 + OC	M10S 02
		Ni	M12N 12
		Steel 2 + OC	M11S 10
		Steel 1	M8S 03
		Single crystal Ni	M7N 02
		Fe-20Cr-2Si	M4S 07
		Fe-16Cr-2Si	M3S 02
	JULI02	Fe-16Cr-2Si	M3S 03
		Fe-20Cr-2Si	M4S 08
		Single crystal Ni	M7N 03
		Steel 1 + OC	M10S 03
		Ni	M12N 03
		Steel 2	M9S11
1.4	JULI03	Cu	M5C 05
		Al + CNT	M2A 12
		Al + CNT	M2A13
		Cu	M5C 06
		Al	M1A 06
		Al	M1A 07
		Cu + CNT	M6C 03
		Cu	M5C 07
		Al + CNT	M2A 15
		Cu + CNT	M6C 07
		Cu + CNT	M6C 08
		Al	M1A 16

		Steel 1	M8S 06
		Steel 2	M9S13
		Steel 1 + OC	M10S 06
		Steel 2 + OC	M11S 19
		Fe-16Cr-2Si	M3S 06
		Fe-20Cr-2Si	M4S 11
		Ni	M12N 06
		Steel 2 + OC	M11S 13
		Steel 1	M8S 07
		Ni + CNT	M13N 03
		Steel 2	M9S14
		Steel 1 + OC	M10S 07
		Ni	M12N 07
		Steel 2 + OC	M11S 14
		Steel 1	M8S 10
		Fe-20Cr-2Si	M4S 12
		Fe-16Cr-2Si	M3S 07
		Ni + CNT	M13N 13
		Fe-16Cr-2Si	M3S 08
		Fe-20Cr-2Si	M4S 13
		Single crystal Ni	M7N 08
		Steel 1 + OC	M10S 16
	JULI04	Steel 2	M9S15
		Ni + CNT	M13N 20
		Ni	M12N 08
		Single crystal Ni	M7N 09
		Single crystal Ni	M7N 10
		Cu	M5C 09
		Al + CNT	M2A 01
		Al + CNT	M2A 02
		Cu	M5C 10
		Al	M1A 11
		Al	M1A 12
		Cu + CNT	M6C 10
		Cu	M5C 11
2.1	JULI05	Al + CNT	M2A 03
		Cu + CNT	M6C 12
		Cu + CNT	M6C 13
		Al	M1A 13
		Steel 1	M8S 15
		Steel 2	M9S18
		Steel 1 + OC	M10S 11
		Single crystal Ni	M7N 11

		Fe-16Cr-2Si	M3S 11
		Fe-20Cr-2Si	M4S 01
		Ni	M12N 17
		Steel 2 + OC	M11S 04
		Steel 1	M8S 16
		Ni + CNT	M13N 07
		Steel 2	M9S19
		Steel 1 + OC	M10S 12
		Ni	M12N 18
		Steel 2 + OC	M11S 05
		Steel 1	M8S 17
		Single crystal Ni	M7N 12
		Fe-20Cr-2Si	M4S 02
		Fe-16Cr-2Si	M3S 12
	JULI06	Ni + CNT	M13N 08
		Fe-16Cr-2Si	M3S 13
		Fe-20Cr-2Si	M4S 03
		Single crystal Ni	M7N 13
		Steel 1 + OC	M10S 13
		Steel 2	M9S20
		Ni + CNT	M13N 09
		Steel 2 + OC	M11S 06
		Ni	M12N 13

6. SUMMARY AND CONCLUSIONS

This report summarizes the disassembly, TM analysis, and future post-irradiation examination of irradiation capsules that contain nanodispersion-strengthened materials for the improved neutron irradiation resistance of fuel cladding and reactor core materials. All six capsules were successfully disassembled, and all TM specimens were shipped to LAMDA for further analysis. The results of this project will support the development of new radiation-resistant materials by helping researchers understand the mechanism of defect evolution at interfaces in nanodispersion-strengthened materials.

7. WORKS CITED

[1] S. Piela, R. Howard, A. Le Coq, K. Linton, and J. Li, *Design and Assembly of Rabbit Capsules for Irradiation of Prototype Metal and Nanocomposite Specimens in the High Flux Isotope Reactor,* ORNL/SPR-2019/1306, Oak Ridge, Tennessee (2019).

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[4] R. Howard and K. Smith, *Development of a Flexible Design for Irradiation of Miniature Tensile and Charpy Test Specimens in the High Flux Isotope Reactor,* ORNL/TM-2018/872, Oak Ridge, Tennessee (2018).

APPENDIX A: INDIVIDUAL SSJ2 SPECIMEN IMAGES

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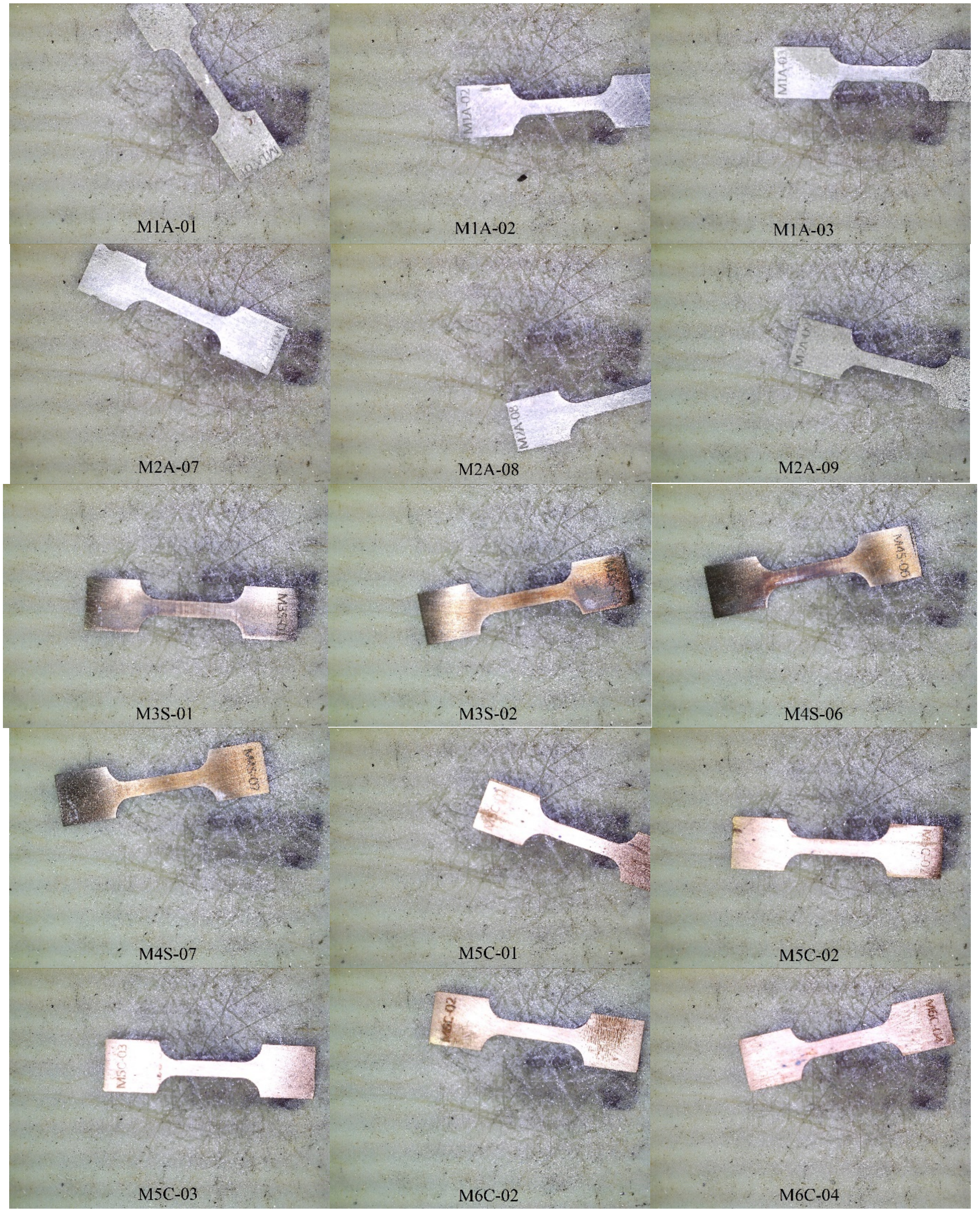
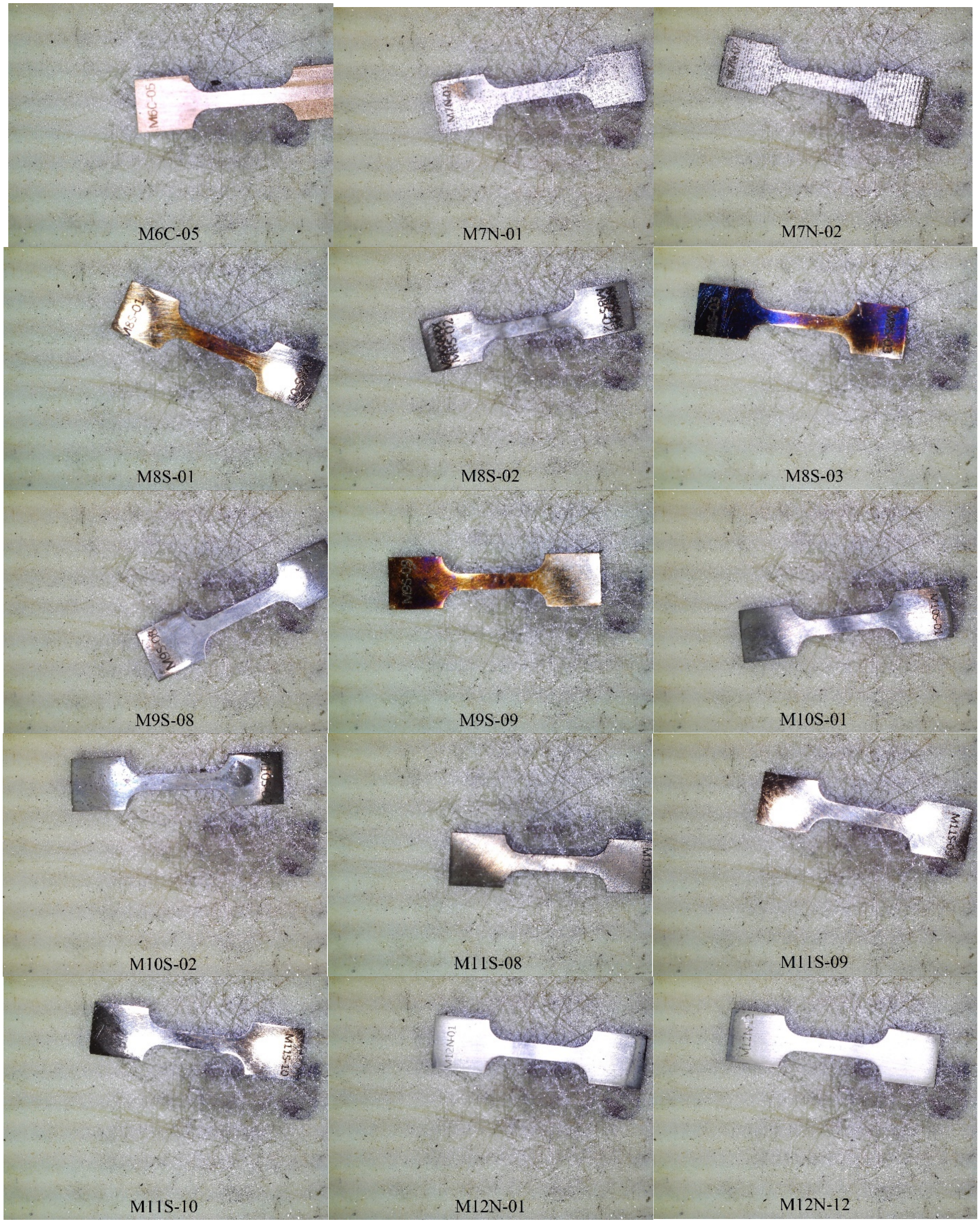
JULI01:

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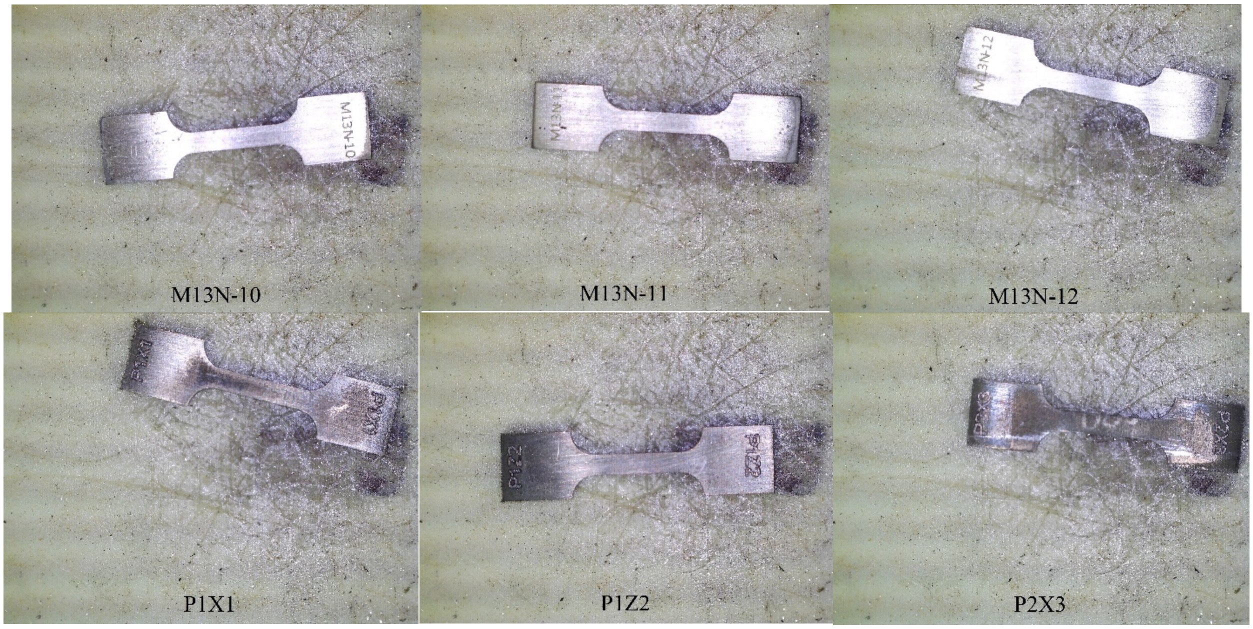
M12N-12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P1Z2
P2X3

JULI02:


FD-01
FD-02
FD-11
M1A-04
M1A-05
M2A-10
M2A-11
M3S-03
M3S-04
M3S-05
M4S-08
M4S-09
M4S-10
M5C-04
M6C-01
M7N-03
M7N-04
M7N-05
M8S-04
M8S-05
M9S-11


M9S-12
M10S-03
M10S-04
M10S-05
M11S-11
M11S-12
M12N-03
M12N-04
M12N-05
M13N-04
M13N-05
M9S-10
P2Z1
P3X2
P3Z3

JULI03:

M1A-06 M1A-07 M1A-16

M2A-12 M2A-13 M2A-15

M3S-06 M3S-07 M4S-11

M4S-12 M5C-05 M5C-06

M5C-07 M6C-03 M6C-07

M6C-08 M7N-06 M7N-07

M8S-06 M8S-07 M8S-10


M9S-13
M9S-14
M10S-06
M10S-07
M11S-13
M11S-14
M11S-19
M12N-06
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M13N-02
M13N-03
P1X2
P1Z3
P2X1

JULI04:


FD-04
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M1A-09
M1A-10
M2A-16
M2A-17
M3S-08
M3S-09
M3S-10
M4S-13
M4S-14
M4S-15
M5C-08
M6C-09
M7N-08
M7N-09
M7N-10
M8S-12
M8S-13
M9S-06


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M10S-09
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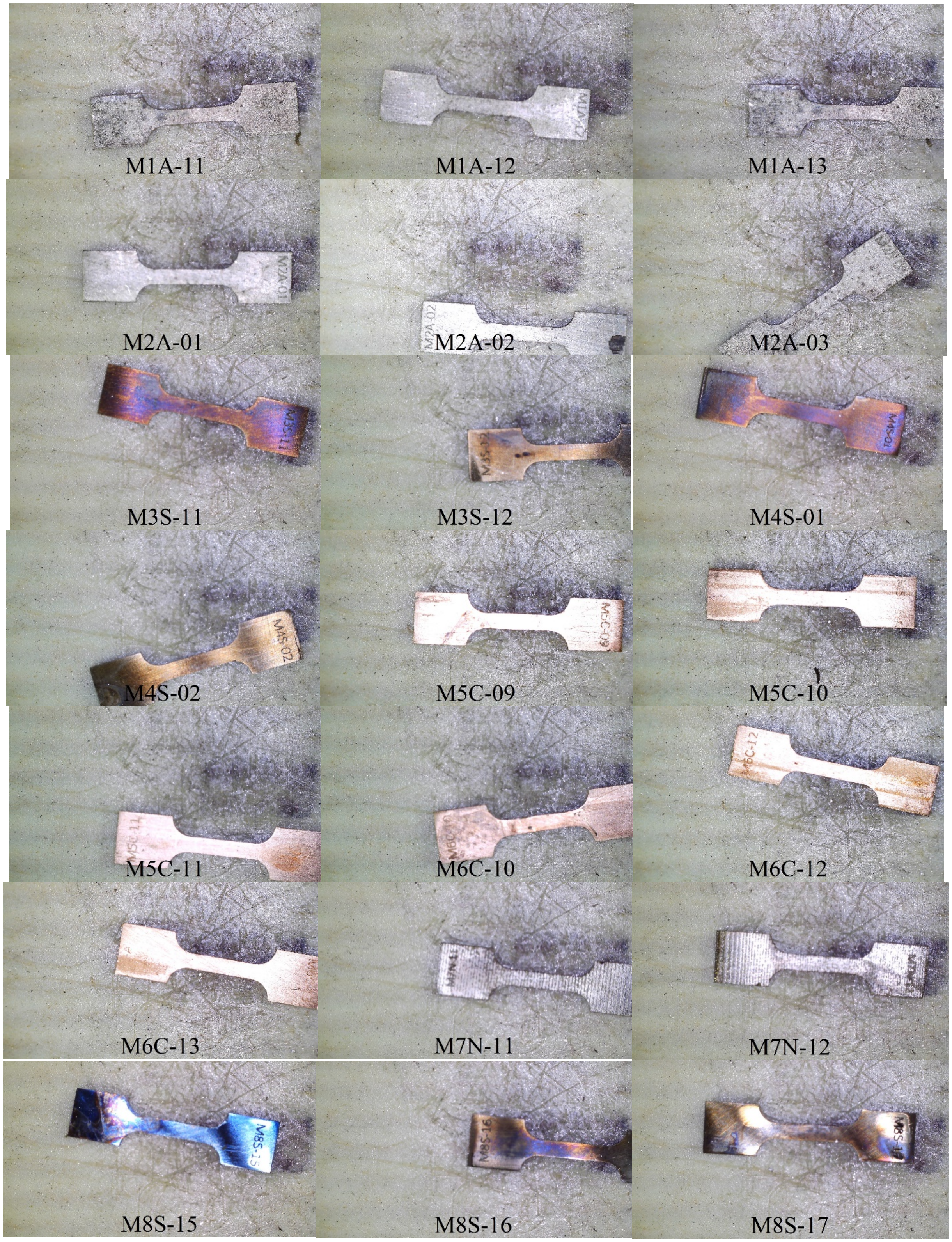
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M8S-16
M8S-17


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M11S-18
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M13N-07
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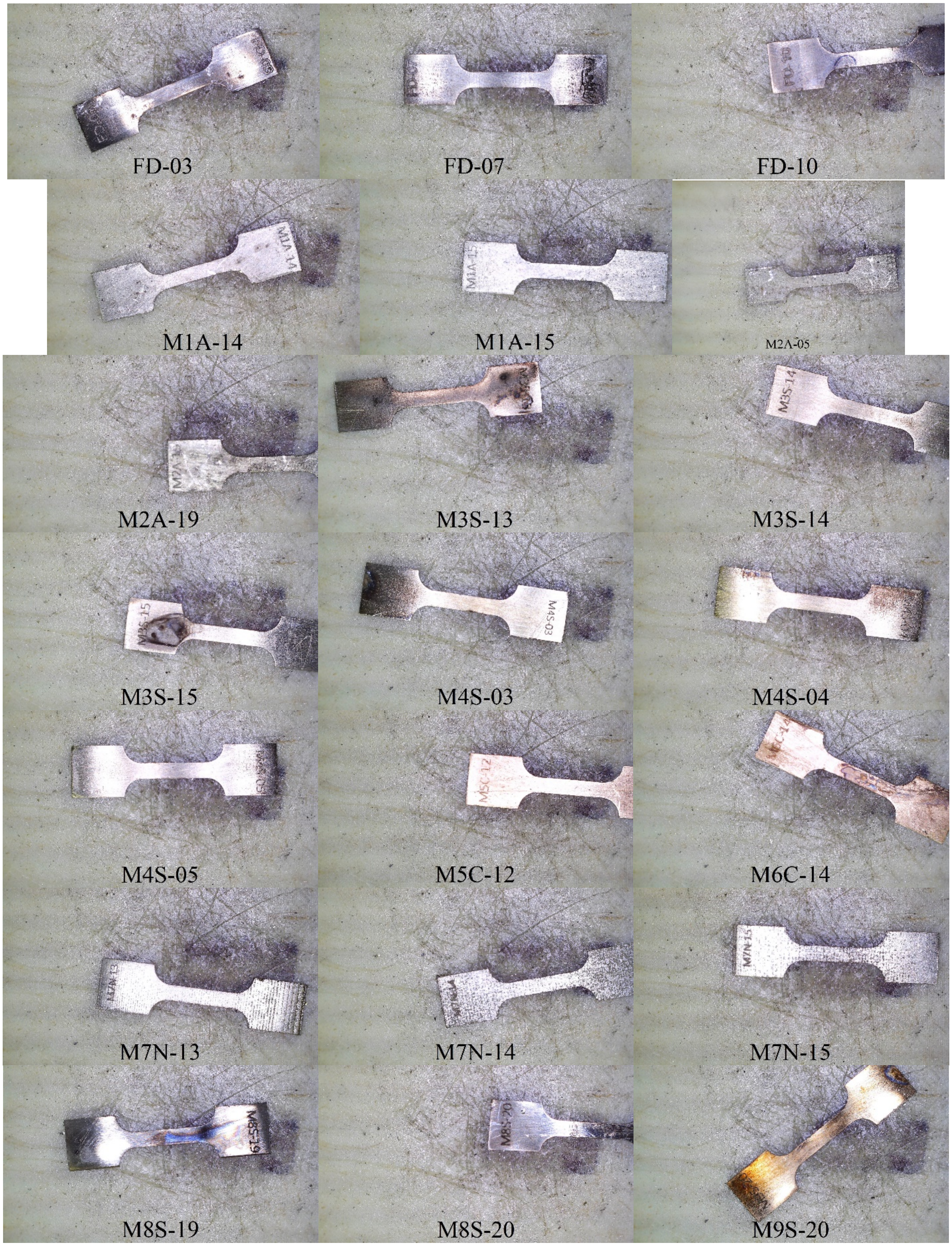
P1Z1
P2X2

JULI06: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M4S-03
M4S-04
M4S-05
M5C-12
M6C-14
M7N-13
M7N-14
M7N-15
M8S-19
M8S-20
M9S-20


M9S-21
M9S-22
M10S-13
M10S-14
M10S-15
M11S-06
M11S-07
M12N-13
M12N-14
M12N-15
M13N-08
M13N-09
P2Z3
P3X1
P3Z2