ORNL/TM-2016/78

Status Report on Irradiation Capsules Containing Welded FeCrAl Specimens for Radiation Tolerance Evaluation

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February 26th, 2016

OAK RIDGE NATIONAL LABORATORY
MANAGED BY UT-BATTELLE FOR THE US DEPARTMENT OF ENERGY

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Nuclear Energy Enabling Technologies (NEET): Reactor Materials

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1. INTRODUCTION

This status report provides the background and current status of a series of irradiation capsules, or "rabbits", that were designed and built to test the contributions of microstructure, composition, damage dose, and irradiation temperature on the radiation tolerance of candidate FeCrAl alloys being developed to have enhanced weldability and radiation tolerance. These rabbits will also test the validity of using an ultra-miniature tensile specimen to assess the mechanical properties of irradiated FeCrAl base metal and weldments. All rabbits are to be irradiated in the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL) to damage doses up to ≥15 dpa at temperatures between 200-550°C.

2. DESCRIPTION OF IRRADIATION CAMPAIGN

FeCrAl alloys are being considered for nuclear power production due to their excellent environmental compatibility in a range of different extreme exposures including liquid metal and aqueous corrosion [1–3]. Given this, a matter of concern based on the previous work done outside the nuclear field is the susceptibility of FeCrAl weldments and weld overlays to cracking using fusion-based welding techniques [4,5]. Furthermore, radiation can lead to a significant change in the overall microstructure in both the base metal and weldments leading to detrimental changes in the mechanical properties with increasing exposure. In order to assess the radiation tolerance of FeCrAl weldments in radiation environments and their susceptibility to cracking, a systematic irradiation campaign that uses a high-flux materials test reactor has been launched. Candidate FeCrAl alloys for the campaign were developed to both reduce the susceptibility of cracking in FeCrAl weldments and increase the radiation tolerance by doing careful modification of the composition and microstructure of the alloys [6–9].

The irradiation campaign has been designed to not only probe the radiation tolerance of different candidate alloys but also to understand the effects of irradiation temperature and damage dose. Three different irradiation temperatures were selected: 200°C, 330°C, and 550°C. Based on previous irradiation studies on Fe-Cr based alloys, these three temperatures will probe three different regimes for the FeCrAl alloys: a dislocation loop dominated regime at 200°C, a mixed dislocation loop and precipitation dominated regime at 330°C, and softening or limited dislocation loop dominated regime at and above 550°C [10]. The selected temperatures also span a wide range of temperatures seen within current fission reactor designs and possible future designs, hence providing critical data needed for the assessment of candidate welded FeCrAl alloys for a variety of nuclear power production applications.

Along with temperature, the irradiation campaign will also investigate the radiation tolerance of the FeCrAl alloys as a function of damage dose. Here, the nominal target doses were selected as 2 dpa, 8 dpa, and 15 dpa. This dose range spans a typical lifetime accumulated dose for a fuel cladding component. Also, doses above ~15 dpa will extend past the expected lifetime of the program and have much larger sample activities limiting possibilities for extensive post-irradiation examination efforts. Based on this design of experiment, the test matrix presented in Table 1 was developed.

Table 1: Test matrix for irradiation within the HFIR. Nominal dose (dpa) is calculated based on a pure-Fe specimen.

HFIR Position	Capsule ID	Target Temp (°C)	Number of Cycles	Nominal Capsule Avg. Flux ($n/cm^2 \cdot s$)	Nominal Capsule Avg. Fluence (n/cm^2)	Nominal Capsule Average Dose (dpa)
PTP D7 5	FCAT01	200	1	$1.10x10^{15}$	$2.81x10^{21}$	1.9
PTP G4 5	FCAT04	200	4	$1.10x10^{15}$	$9.12x10^{21}$	7.6
PTP G7 5	FCAT07	200	8	$1.10x10^{15}$	$1.79x10^{22}$	15.2
PTP D7 6	FCAT02	330	1	$1.08x10^{15}$	$2.24x10^{21}$	1.9
PTP G4 6	FCAT05	330	4	$1.08x10^{15}$	$8.96x10^{21}$	7.4
PTP G7 6	FCAT08	330	8	$1.08x10^{15}$	$1.79x10^{22}$	14.9
PTP D7 5	FCAT03	550	1	$1.10x10^{15}$	$2.81x10^{21}$	1.9
PTP G4 5	FCAT06	550	4	$1.10x10^{15}$	$9.12x10^{21}$	7.6
PTP G7 5	FCAT09	550	8	$1.10x10^{15}$	$1.79x10^{22}$	15.2

To reach the desired test temperatures and damage doses listed in Table 1 using the HFIR, small irradiation capsules, or “rabbits”, are used which can house a varying range of different specimens including sub-sized tensile specimens. Two different tensile geometries were selected, one being a more “normal” SS-J type configuration and the recently developed SS-2E ultra-miniature tensile specimen. A schematic of the tensile specimens is shown in Figure 1. This irradiation campaign will be the first set of experiments assessing the validity of using SS-2E geometries to develop a mechanical properties database on irradiated FeCrAl base alloys and weldments. The SS-2E geometry is an attractive design to use as it significantly reduces the activated volume of a macro-scale tensile specimen with limited sacrifice in base mechanical properties [6,7,9]. As such, it provides greater flexibility in post-irradiation experiments and could garner more details on the deformation mechanics of irradiated FeCrAl alloys.

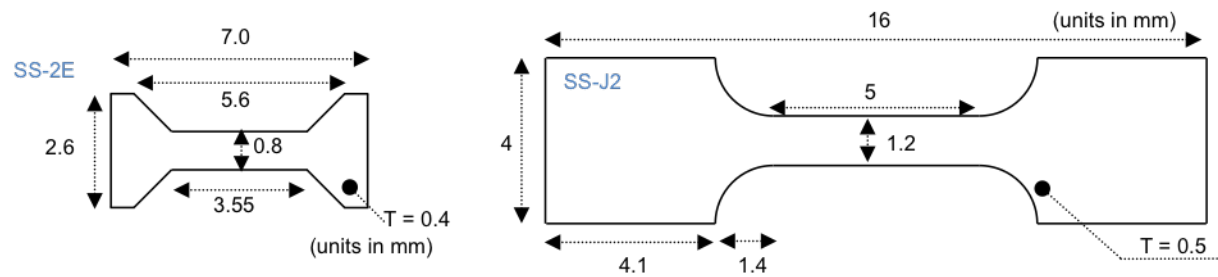

Figure 1: Simplified schematic of the tensile specimen geometries for weld and irradiation testing. Figure not to scale. Reproduced from Ref. [8].

The rabbits were designed in a way to optimize the number of SS-J type (27 specimens) and SS-2E (18 specimens) in a single configuration. The rabbits were also designed to include 12 (4 radially x 3 positions axially) passive SiC thermometry specimens to validate the modeled target irradiation temperatures. To ease post-irradiation examination and extraction of the specimens from the rabbits, samples were placed into three separate sub-housing or modules within the primary outer containment. A three-dimensional rendering of the design is provided in Figure 2.

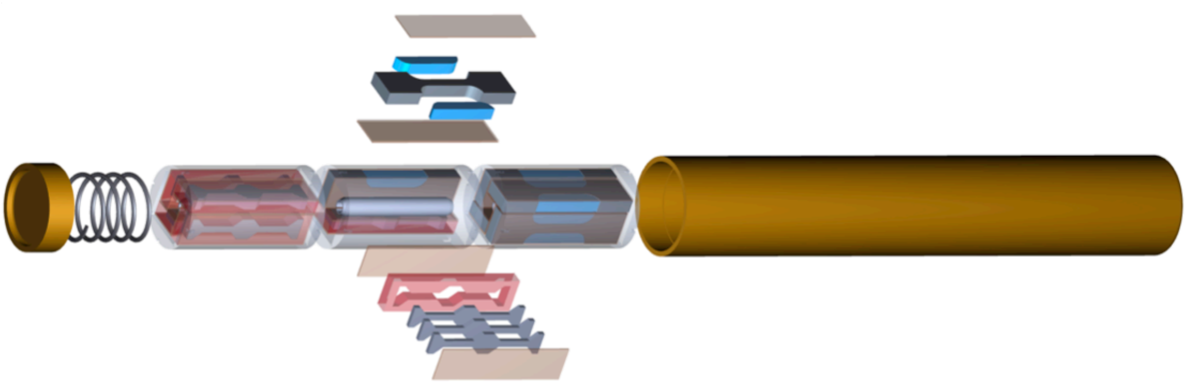

Figure 2: Finalized HFIR rabbit design for irradiation of welded and non-welded tensile specimens.

Temperature of the rabbit design selected in Table 1 is controlled by the axial location within the HFIR, the selected fill gas (typically He), and the radial gap between the specimen holder (transparent geometries in Figure 2) and the outer housing. For this irradiation campaign, He was selected as the back fill gas while the axial positions provided in Table 1 for HFIR were selected as they reside near the HFIR core centerline resulting in limited variation in axial flux and hence minimizing thermal gradients axially. The result is the holder diameter (and material) was the primary variable for meeting the design temperatures in Table 1. The resulting modeled temperatures, associated HFIR positions, and holder diameters are provided in Table 2.

Table 2: Thermal analysis conditions for a mixed SS-2E and SS-J2 tensile specimens' rabbit.

Target Temperature	HFIR Position	Holder Diameter	Specimen Location	Specimen Analysis Conditions		
				Avg.	Min.	Max.
200°C	PTP 5	9.43 mm	Outer	199°C	171°C	242°C
			Middle	222°C	177°C	263°C
			Inner	235°C	181°C	277°C
330°C	PTP 6	9.28 mm	Outer	314°C	288°C	351°C
			Middle	336°C	292°C	373°C
			Inner	348°C	300°C	386°C
550°C	PTP 5	9.00 mm	Outer	502°C	476°C	543°C
			Middle	525°C	484°C	563°C
			Inner	537°C	488°C	575°C

The tensile specimens shown in Figure 1 and Figure 2 were fabricated from sheet feedstock of varying FeCrAl compositions and microstructures developed within this program. Details on the alloys have been described in greater detail and depth elsewhere [6,9]. Eighteen different unique sample configurations (alloy type, sample type, and welded or non-welded) were prepared for irradiation, see Table 3 for the single rabbit loading lists. It should be noted several alloys not of direct interest in this program are included to enable cross comparison of different alloy concepts and provide better scientific understanding of several different possible degradation mechanisms in the FeCrAl alloys. Here, only 2-3 tensile specimen types per specimen type are used in a single irradiation rabbit. This will allow for some repeatability in the data to be assessed. It is recognized that more samples would provide better statistics but a balance between assessing different specimens and statistics was struck in the current loading configurations.

Table 3: Single rabbit loading list by alloy type, specimen type, and geometry. Alloys FCA-ODS and C06M are FeCrAl alloys currently of interest in other irradiation programs.

Material Code	Condition	Number of SS-J2 per rabbit	Number of SS-2E per rabbit
C35M	non-welded	3	2
C36M	non-welded	3	-
C37M	non-welded	3	2
C06M	non-welded	3	-
FCA-ODS	non-welded	2	-
C35MN	non-welded	2	2
C35M10TC	non-welded	3	2
C35M	welded	2	2
C37M	welded	2	3
C35MN	welded	2	2
C35M10TC	welded	2	3

All samples were manufactured from a single outside vendor in an effort to limit differences due to variances in manufacturing practices. After receiving the samples from the vendor, all samples were cleaned and visually inspected. Samples which did not meet the rigorous quality standards of the program were removed from the sample set for irradiation. Each sample was laser etched to provide a unique sample identity. Identification marks were placed on both tensile heads to keep traceability after fracture during mechanical testing. The laser marking scheme can be found in Table 4.

Table 4: Specimen ID marking for specimens for weld and irradiation testing

Spec ID.	Specimen Type	Material Code	Condition
MFXX	SS-J2	C35M	non-welded
MVXX6	SS-J2	C36M	non-welded
MVXX	SS-J2	C37M	non-welded
THXX	SS-J2	C35MTC10	non-welded
N5XX	SS-J2	C35MN	non-welded
FXX	SS-2E	C35M	non-welded
IXX	SS-2E	C37M	non-welded
HXX	SS-2E	C35M10TC	non-welded
NXX	SS-2E	C35MN	non-welded

Spec ID.	Specimen Type	Material Code	Condition
MFXXW	SS-J2	C35M	welded
VWXX	SS-J2	C37M	welded
NWXX	SS-J2	C35MN	welded
HWXX	SS-J2	C35M10TC	welded
AXX	SS-2E	C35M	welded
BXX	SS-2E	C35MN	welded
SXX	SS-2E	C37M	welded
OXX	SS-2E	C35M10TC	welded

Prior to loading into the rabbits, care was taken to record the dimensions of each specimen including the gauge width and height to allow for proper calculation of tensile curves after irradiation. Samples were then loaded into the sub assemblies and outer housing. Images of the FCAT01 rabbit during sample loading are shown in Figure 3 and Figure 4. A cartoon was used to record the location of each specimen within the rabbit enabling cross-correlation between sample location and determined irradiation temperature in each sub-assembly. Final cartoons providing the location for the first three rabbits (FCAT01, FCAT02, and FCAT03) are provided in Appendix A. After loading, rabbits were initially sealed with a circumferential electron beam weld followed by leak testing to verify the quality of the weld. Rabbits were then back-filled with He and the weeping hole that allowed for gas communication and backfill into the rabbit was sealed using a pulsed laser beam weld. Final inspection included hydrostatic testing to ensure the overall quality of the welds and structural performance of the rabbit prior to insertion into the HFIR. Figure 5 shows the first three completed rabbits immediately prior to insertion in the HFIR core.

Figure 3: Internal components of the FCAT01 capsule during specimen loading.

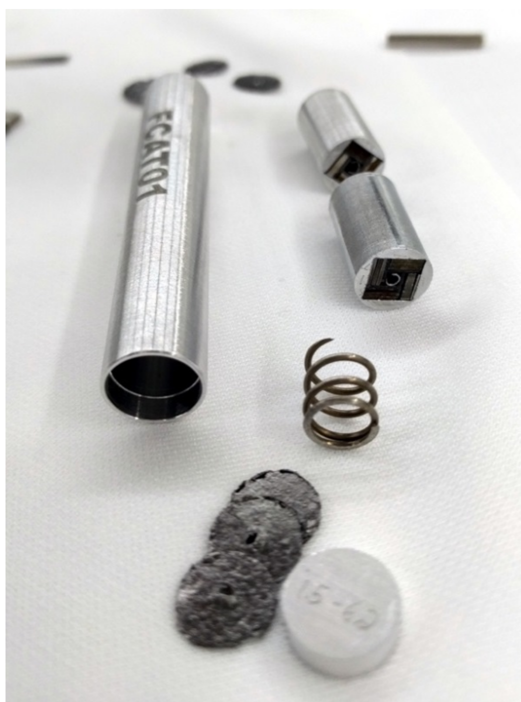

Figure 4: Photograph of the FCAT01 rabbit during specimen loading.

Figure 5: Photograph of completed rabbits prior to loading in the HFIR core.

3. STATUS OF IRRADIATION CAMPAGIN

The number of cycles required per rabbit for the desired fluence is provided in Table 1. The first three rabbits (FCAT01, FCAT02, and FCAT03) were inserted in the HFIR core on February 12, 2016 and started irradiation at 85 MW in cycle 465 on February 23, 2016. These three capsules will be irradiated for one cycle and be removed from the HFIR core on March 18, 2016 resulting in a total irradiation time of 24 days. Following irradiation, the capsules and corresponding samples will be held at HFIR for a period of approximately thirty days to allow for short-lived radioisotopes to decay and reduce the overall radiological threat of the capsules prior to shipment to the Irradiated Materials Examination and Testing (IMET) facility at ORNL. Therefore, post irradiation examination of the low dose capsules will begin in mid-April to May 2016. After capsule opening and sample sorting in IMET, specimens will be shipped to the Low Activation Materials Development and Analysis (LAMDA) facility for extensive mechanical and microstructural examination.

The remaining mid-dose and high-dose capsules (FCAT 04-09) will be inserted into the HFIR core during the cycle 465 outage scheduled to start on March 18, 2016 and finish on May 31, 2016. Irradiation of these capsules will begin starting on cycle 466 on May 31, 2016. The mid-dose capsules will be irradiated for four cycles and finish irradiation on December 16, 2016. The highest dose capsules will run for eight cycles, current HFIR operating schedule and forecasting does not provide details for the last two cycles, but is anticipated the samples will finish irradiation within the second quarter of the US fiscal year 2017. A breakdown of expected dates for all rabbits is provided in Table 5.

Table 5: Anticipated irradiation campaign schedule. Dates are approximant.

Capsule ID	Target Temp (°C)	Number of Cycles	Average Capsule Dose (dpa)	Capsule Insertion Date	Capsule Release Date	Cycle Run Numbers	Shipment Date to IMET
FCAT01	200	1	1.9	2/23/16	3/18/16	465	4/18/16
FCAT04	200	4	7.6	5/31/16	12/16/16	466-469	1/16/17
FCAT07	200	8	15.2	5/31/16	TBD	466-474	TBD
FCAT02	330	1	1.9	2/23/16	3/18/16	465	4/18/16
FCAT05	330	4	7.4	5/31/16	12/16/16	466-469	1/16/17
FCAT08	330	8	14.9	5/31/16	TBD	466-474	TBD
FCAT03	550	1	1.9	2/23/16	3/18/16	465	4/18/16
FCAT06	550	4	7.6	5/31/16	12/16/16	466-469	1/16/17
FCAT09	550	8	15.2	5/31/16	TBD	466-474	TBD

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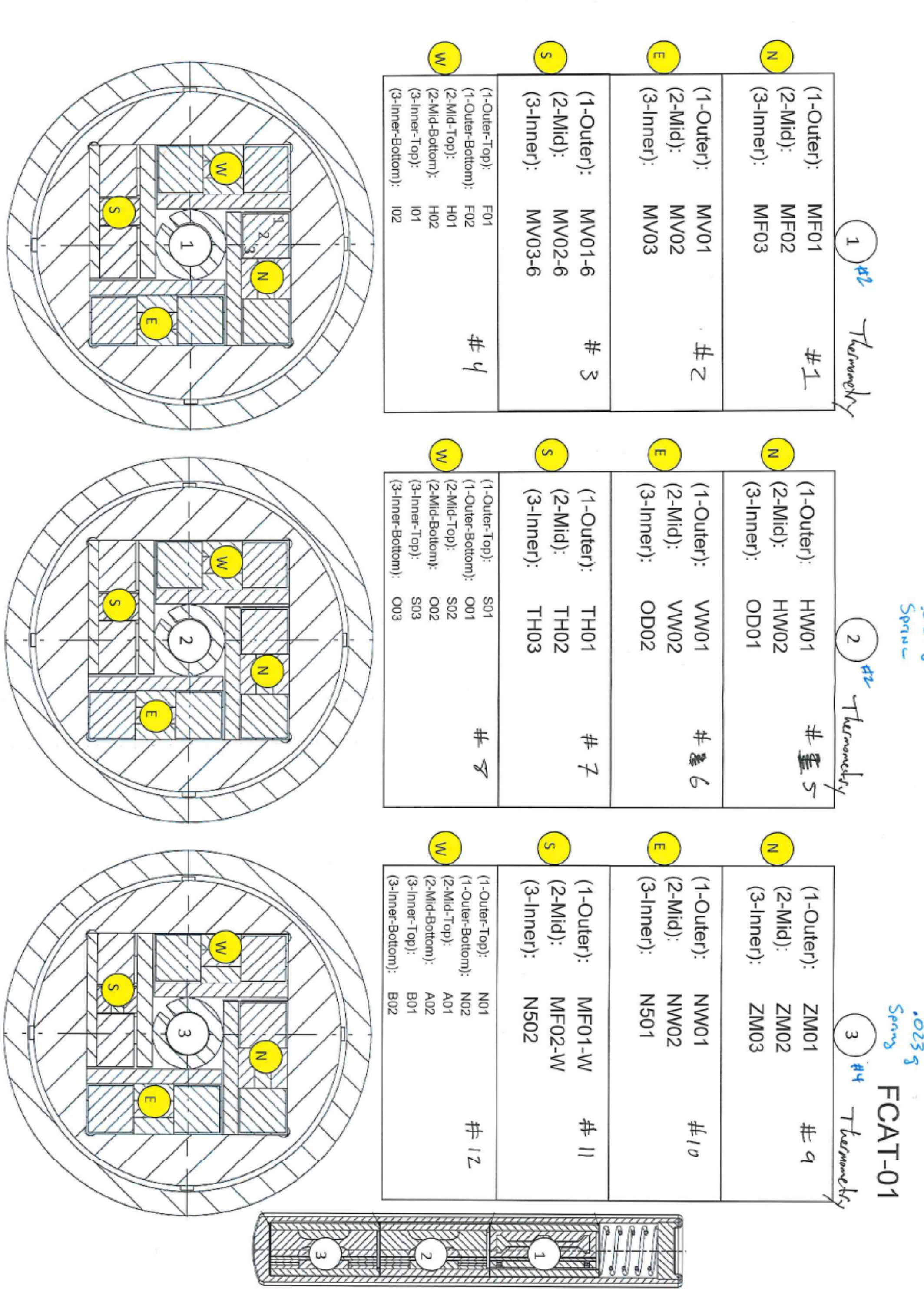
APPENDIX A: LOADING CARTOONS FOR LOW DOSE RABBITS



FCAT-02

1 — .023g Spring — Thermometry

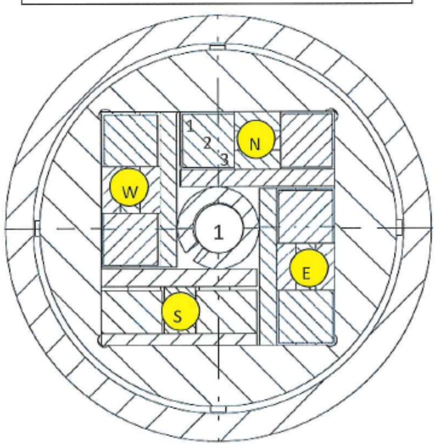
			Thermometry
N	(1-Outer):	MF04	13
	(2-Mid):	MF05	
	(3-Inner):	MF06	
E	(1-Outer):	MV04	14
	(2-Mid):	MV05	
	(3-Inner):	MV06	
S	(1-Outer):	MV04-6	15
	(2-Mid):	MV05-6	
	(3-Inner):	MV06-6	
W	(1-Outer-Top):	F03	16
	(1-Outer-Bottom):	F04	
	(2-Mid-Top):	H03	
	(2-Mid-Bottom):	H04	
	(3-Inner-Top):	I03	
	(3-Inner-Bottom):	I04	

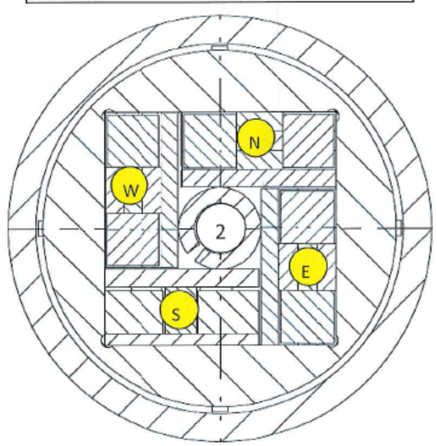
2 — .023g Spring — Thermometry

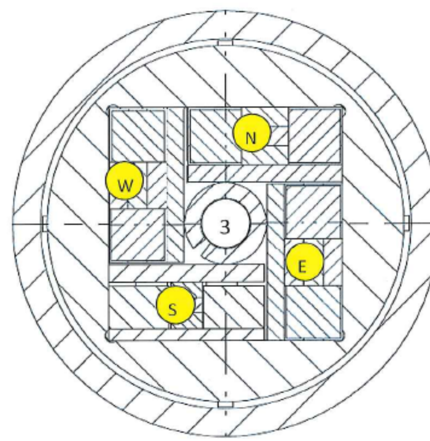
			Thermometry
N	(1-Outer):	HW03	17
	(2-Mid):	HW04	
	(3-Inner):	OD03	
E	(1-Outer):	VW03	18
	(2-Mid):	VW04	
	(3-Inner):	OD04	
S	(1-Outer):	TH04	19
	(2-Mid):	TH05	
	(3-Inner):	TH06	
W	(1-Outer-Top):	S04	20
	(1-Outer-Bottom):	O04	
	(2-Mid-Top):	S05	
	(2-Mid-Bottom):	O05	
	(3-Inner-Top):	S06	
	(3-Inner-Bottom):	O06	

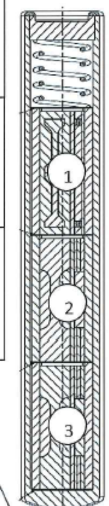
3 — .023g Spring — Thermometry

			Thermometry
N	(1-Outer):	ZM04	21
	(2-Mid):	ZM05	
	(3-Inner):	ZM06	
E	(1-Outer):	NW03	22
	(2-Mid):	NW04	
	(3-Inner):	N503	
S	(1-Outer):	MF03-W	23
	(2-Mid):	MF04-W	
	(3-Inner):	N504	
W	(1-Outer-Top):	N03	24
	(1-Outer-Bottom):	N04	
	(2-Mid-Top):	A03	
	(2-Mid-Bottom):	A04	
	(3-Inner-Top):	B03	
	(3-Inner-Bottom):	B04	









FCAT-03

1 (Thermometry)

			Thermometry
N	(1-Outer):	MF07	25
	(2-Mid):	MF08	
	(3-Inner):	MF09	
E	(1-Outer):	MV07	26
	(2-Mid):	MV08	
	(3-Inner):	MV09	
S	(1-Outer):	MV07-6	27
	(2-Mid):	MV08-6	
	(3-Inner):	MV09-6	
W	(1-Outer-Top):	F05	28
	(1-Outer-Bottom):	F06	
	(2-Mid-Top):	H05	
	(2-Mid-Bottom):	H06	
	(3-Inner-Top):	I05	
	(3-Inner-Bottom):	I06	

2 (Thermometry)

			Thermometry
N	(1-Outer):	HW05	29
	(2-Mid):	HW06	
	(3-Inner):	OD05	
E	(1-Outer):	VW05	30
	(2-Mid):	VW06	
	(3-Inner):	OD06	
S	(1-Outer):	TH07	31
	(2-Mid):	TH08	
	(3-Inner):	TH09	
W	(1-Outer-Top):	S07	32
	(1-Outer-Bottom):	O07	
	(2-Mid-Top):	S08	
	(2-Mid-Bottom):	O08	
	(3-Inner-Top):	S09	
	(3-Inner-Bottom):	O09	

3 (Thermometry)

			Thermometry
N	(1-Outer):	ZM07	33
	(2-Mid):	ZM08	
	(3-Inner):	ZM09	
E	(1-Outer):	NW05	34
	(2-Mid):	NW06	
	(3-Inner):	N505	
S	(1-Outer):	MF05-W	35
	(2-Mid):	MF06-W	
	(3-Inner):	N506	
W	(1-Outer-Top):	N05	36
	(1-Outer-Bottom):	N06	
	(2-Mid-Top):	A05	
	(2-Mid-Bottom):	A06	
	(3-Inner-Top):	B05	
	(3-Inner-Bottom):	B06