

THE MONBUSHO/U.S. SHIELDED HFIR IRRADIATION EXPERIMENT: HFIR-MFE-RB-11J AND 12J (P3-2 AND P3-3) — M. L. Grossbeck, K. E. Lenox, and M. A. Janney (Oak Ridge National Laboratory), T. Muroga (National Institute for Fusion Science), D. W. Heatherly and K. R. Thoms (Oak Ridge National Laboratory)

SUMMARY

This experiment is a joint project between the Japanese Monbusho, the Japan Atomic Energy Research Institute, and the U.S. Fusion Energy Sciences Program. It is the first of a series of experiments using europium oxide as a thermal neutron shield to minimize transmutations in vanadium alloys and ferritic/martensitic steels. The europium oxide shields were developed using ceramic processing techniques culminating in cold pressing and sintering. This experiment, which is a prototype for future fast neutron experiments in the HFIR, contains approximately 3200 specimens of 18 different types. The experiment began operating at 300 and 500°C in February 1997 and is projected to attain its goal fluence of ~5 dpa in February 1998.

PROGRESS AND STATUS

Introduction

This experiment is a low exposure irradiation containing a large variety of specimens primarily for microstructural analysis and mechanical properties of a wide variety of materials. It consists of two irradiation vehicles, one at 300°C (HFIR-MFE-RB-11J) and the other at 500°C (HFIR-MFE-RB-12J). Vanadium transmutes to chromium by thermal neutron absorption with an absorption cross section of 80 barns. As a result, initially pure vanadium will contain 10% Cr after an exposure of 5 dpa in a HFIR reflector position. For the low activation ferritic-martensitic steels, transmutation of tantalum and tungsten, which have thermal absorption cross-sections of 25 and 19 barns, respectively, could significantly perturb alloy composition. To minimize these transmutation effects, a europium oxide shield was developed for this experiment that reduces the thermal flux by a factor of about 100.

To maintain controlled specimen temperatures and minimize radiative heat transfer, the specimens are held in carefully designed racks of aluminum alloy. The specimens had to be machined to close tolerances in order to fit into the racks and to achieve good thermal contact. Twenty-one thermocouples are positioned along the length of each capsule, some near the centerline, and the remainder toward the periphery of the holders. Temperature is maintained by surrounding the specimen chamber with an annulus of inert gas which can be varied in composition from pure helium to neon or argon, or a mixture of helium and one of the other gases. An automatic computer-controlled system uses an average thermocouple temperature for control and adjusts the gas mixture to maintain the design temperature.

Experiment Goals

The irradiation vehicles contain many varied experiments for fusion materials research. Only a few examples will be stated. Pressurized tubes of low Cr-Ti-vanadium alloys are being irradiated for the first time to evaluate irradiation creep in this alloy class. Pressurized tubes of ferritic/martensitic alloys are also included to evaluate the newer low-activation ferritic/martensitic steels. A large portion of the space is devoted to ceramic materials, including ceramic/metalllic braze joints. Advanced SiC/SiC fiber composites using low-oxygen SiC are being irradiated to study irradiation-induced densification. Alumina and silicon nitride bend bars of different thicknesses are included to study size effects on fracture of irradiated ceramics. There is also a series of TEM disks with varying minor element chemistry to study the effects of transmutation on microstructure. Mechanical test samples of tensile, compact tension, and Charpy bars as well as bend bars are included to evaluate mechanical properties of several classes of alloys and ceramics. Table 1 presents the specimen loading by material and type of specimen.

Table 1. Specimen Loading for HFIR-MFE-RB-11J

Table 1. Specimen Loading for HFIR-MFE-RB-11J (cont'd)

Alloy	PRZ Tubes (JP)	1.5 mm C/N (JP)	SS-J (JP)	TEM (JP/ US)	DGT (JP/ US)	U.S. SS-3	U.S. PCVN	U.S. CVN	Specimen Loading for HFIR-MFE-RB-11J (300°C)			
									Bend Bar 1 2x2x15mm (JP)	Bend Bar 2 1x2x15 (JP)	Bend Bar 3 0.5x2x15 (JP)	Bend Bar 4 .25x2x15 (JP)
Cr/Cu Alloys												
V/Ceramics												
SiC Fiber												
Transmutation Sedas												
V						6/0						
V-X-Cr						6	30/15					
V-4Ti-4Cr						6	40/20					
Cu						6	40/20					
Cu-xNi-yZnNi						4	20/20					
Cu-xNi-yZnNi						4	40/20					
Fe						6	40/20					
Fe/W&Mn						6	30/15					
OLS						6	20/10					
Fe-Cr-Ni						6	30/15					
Fe-Cr-Ni-X						6	40/20					
316Ti/TiN						6	30/15					
TiAl							10/5					
SiC							20/10					
Al-Al-Si							20/10					
Mo Alloys							20/10					
Irradiated Cu alloys							9/0					
JAERI/U.S.												
F82H(HT-1)							6	10	12	6	5	
F82H(HT-2)							2	10	8	5	5	
F82H Ni doped							2	10	5	5		
F82H Yield							2	10	4	5		
F82H Yieldment							2	4	4	5		
F82H-HAZ							2	10	6	5	5	
9Cr-2WVta							2	10	6	5		
Ferritic Alloys TBD							50					

2 Temp Monitors
(Same as T1)

Table 1. Specimen Loading for HFR-MFE-RB-12.1

Alloy	PRZ Tubes JP/US	1.5mm CVN (JP)	SS-J (JP)	DCT (JP/ US)	TEM (JP/ US)	U.S. CVN	U.S. SS-3	500°C					Thermal Diffusivity 100x2mm (US)	
								Bend Bar 1 2x2x15mm (JP)	Bend Bar 3 0.5xx15 (JP)	Bend Bar 2 1x2x15 (JP)	Bend Bar 4 .25x2x15 (JP)	Bend Bar 5 .6x1.4x9 (JP)	Bend Bar 6 2x4x25 (PNL/ORNL)	Bend Bar 7 0.7x1x10 (JP)
JLF-1	5/0	6	3	3/3	15/0	10	5	5						
JLF-1B	3/0	6	3		6/0									
JLF-1Mn1	3/0	6	2		6/0									
JLF-1Mn2	3/0	6	2		6/0									
JLF-1S														
JLF-1LM														
J.F-1LSM														
F&H1	5/0	6	2		10/0									
Kimura (Ferritic)					3/0/0									
ODSHK (Ferritic)					6/0									
9Cr-2WVTA														
V-4Cr-4Ti-1Si(JP)					9		10/0							
V-4Cr-4Ti(JP)					9		10/0							
V-5Cr-4Ti(JP)					9		10/0							
V-4Cr-3Ti(JP)					9		10/0							
V-5Cr-3Ti(JP)					9		10/0							
V-5Cr-5Ti(JP)					9		10/0							
V-4Fe-4Ti-1Si					9		10/0							
V-4Fe-4Ti					9		10/0							
V-4Fe-3Ti					9		10/0							
V-4Cr-4Ti-1SiR					9		10/0							
V-4Cr-4Ti-SiAlYX					10		5/0/0							
V-4Cr-4Ti 832865	0/6					0/3	3	5						
(Partial rd)														
V-4Cr-4Ti 832865						0/3	3							
(Full rd)														
V-5Cr-5Ti						0/4	1							
V-4Cr-4Ti-Si						0/4	1							
V-3Cr-3Ti						0/4	2							
V-6Cr-3Ti						0/4	2							
V-6Cr-6Ti						0/4	2							
V-4Cr-4Ti-B						0/4	2							
SIC/SiC												24		
SiC (B,B ₂ N)												9		
Al ₂ O ₃												10		
AlN												16		
MgO/Al ₂ O ₃												9		
SiO ₂												3		
TiCx												9/0		

1 Temp Monitors
(Same as II)

Table 1. Specimen Loading for HFIR-MFE-RB-12J (cont'd)

Specimen Loading Configuration

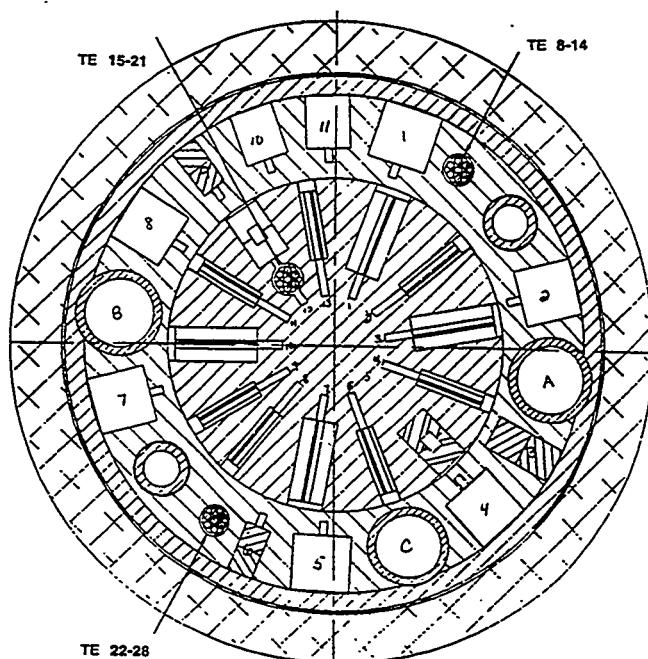
The specimens are loaded into round holes and square or rectangular slots along the longitudinal axes of the capsules. Figure 1 shows the center sections of the capsules identifying the holes and slots. The upper and lower sections of each capsule are shown in Fig. 2. In HFIR-MFE-RB-11J, which will be referred to as 11J, the upper and lower sections contain disc compact tension specimens (DCT). In HFIR-MFE-RB-12J, which will be referred to as 12J, the upper and lower sections contain Charpy v-notch specimens (CVN), bend bar specimens (BB), and DCT specimens. The overall geometry of the specimens is shown in Table 2.

Table 2. Specimen Types and Overall Sizes

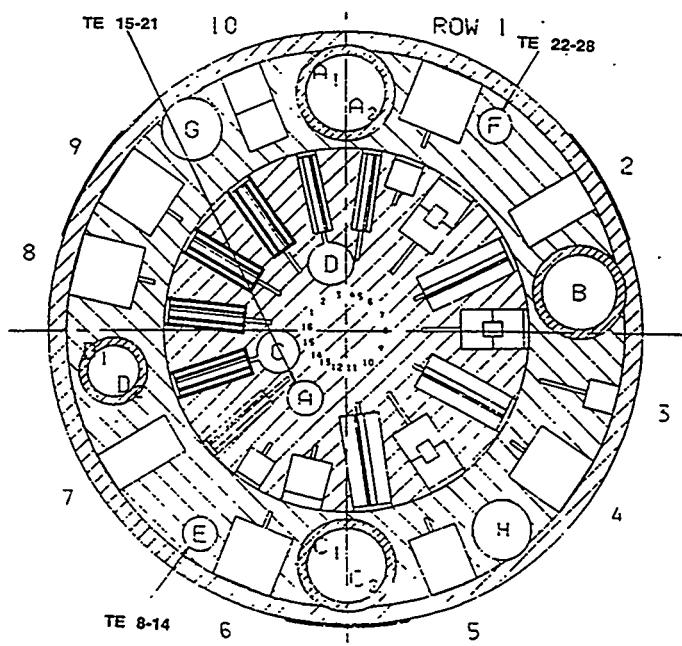
(The experimenter is the person who may be contacted to obtain drawings.)

Specimen Type	Designation	Overall Dimensions (mm)	Experimenter
Trans. Elect. Micros.	TEM	3 x 0.15-0.25	
Tensile	SSJ	16 x 4 x 0.25	Kohno
Tensile	SS-3	25 x 4.95 x 0.76	Grossbeck
Pressurized Tube		22.4 x 4.57 dia	Kohno
Compact Tension	DCT	9.6 dia x 3.93	Kimura
Compact Tension	DCT	12.5 dia x 4.62	Grossbeck
Charpy	1.5 CVN	20 x 1.5 x 1.5	Kohno
Charpy	CVN	25 x 3.33	Grossbeck
Bend Bar	BB1	15 x 2 x 2	Satou
Bend Bar	BB2	15 x 2 x 1	Satou
Bend Bar	BB3	15 x 2 x 0.5	Satou
Bend Bar	BB4	15 x 2 x 0.25	Satou
Bend Bar	BB5	9.0 x 1.4 x 0.6	Satou
Bend Bar	BB6	25 x 4 x 2	Satou
Bend Bar	BB7	10 x 1 x 0.7	Satou
Bend Bar	BB8	25 x 3 x 2.5	Snead
Fiber Tube	SiC Tube	48 x 2 dia	Satou
Thermal Cond.	TD	10 dia x 2	Satou

The specimen loading for the upper and lower sections of 11J is shown in Table 3. Tables 4 and 5 contain the specimen loading for 12J. Since specimens are loaded side by side in a given slot, two columns are allotted for each row (slot). As can be seen in Fig. 1, there is an inner and an outer specimen holder in the center section of each capsule. The two holders are separate and fit concentrically together very tightly so as not to impede radial heat transfer. The specimen loading for the 11J inner holder is given in Table 6. The specimens are listed top to bottom as

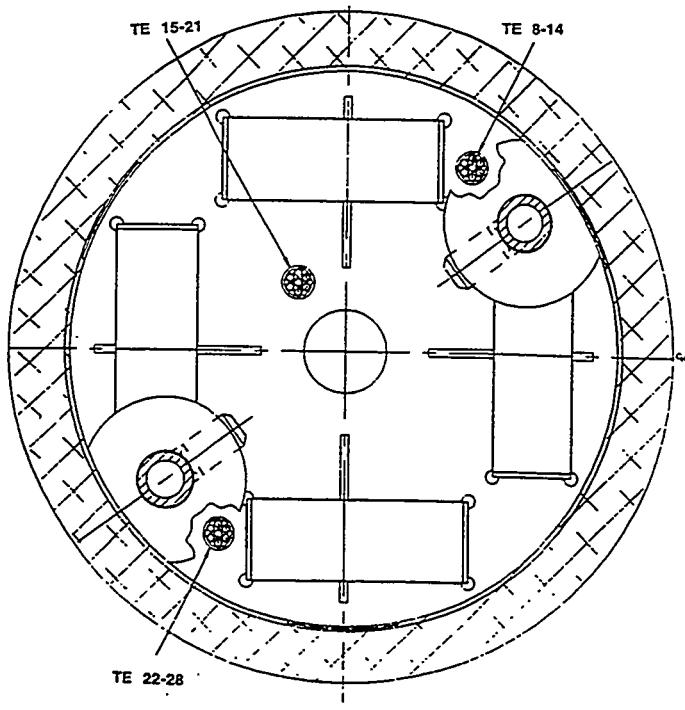


(A) HFIR-MFE-RB-11J

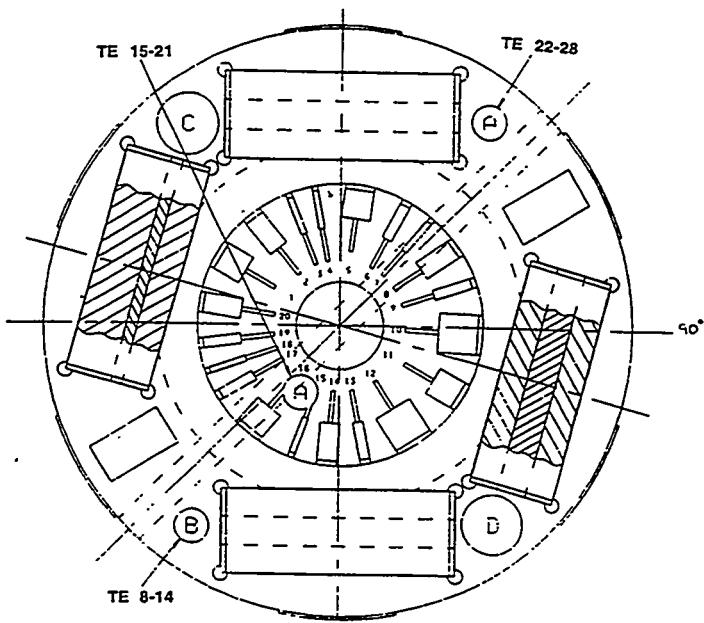


(B) HFIR-MFE-RB-12J

Fig. 1. Cross section through the central portion of the irradiation capsules indicating the designations for the holes and slots.



(A) HFIR-MFE-RB-11J



(B) HFIR-MFE-RB-12J

Fig. 2. Cross section through the end sections of the irradiation capsules.

Table 3. Specimen Loading for the End Sections of HFIR-MFE-RB-11J

Upper Section				
Specimen Type	DCT	DCT	DCT	DCT
Row	1	2	3	4
Z106	LFV5	N202	FA13	
FA01	LFV4	TJO2	FA12	
M1	LFV3	TWO2	FA11	
Lower Section				
TWO1	LFW2	LFV2	FA10	
TJ01	LFW1	LFV1	FA09	
N201	FA00	Z105	FA08	

Table 4. Specimen Loading for the Upper Section of HFIR-MFE-RB-12J

Table 5. Specimen Loading for the Lower Section of HFIR-MFE-RB-12J

Lower Section	DCT	BB6	DCT	DCT	DCT	BB6	1.5 CVN, BB1-BB4
Row	1	2	3	4	Inner		
LFTF	D3	LFTH	Spacer	FA16	Spacer	FA19	LHW1
LFTD		LFTG		FA15		FA18	DY60
LFTA		LFTB		FA14		FA17	DY74
							DY75
							LDW1
							DY28
							DY79
							DY63
							DY64
							DY42
							LDW3
							DY51
							DT66
							DY61
							DY76
							LDW2
							DY67
							DY65
							DY62

Table 6. Specimen Loading for the Central Inner Holder for HFIR-MFE-RB+1J

Container Sustainer	Inner Holder													
Row		1	2	3	4	5	6	7						
Specimen Type ...		SS-3	SS-J	SS-3	SS-3	SS-4	BB5, BB7	SS-J						
F	FA12	FA11	AV52	AV48	70	70	LFV6	LFV7	D--S	F44R	TJ03	TJ04		
F	FA09	FA10	F24G	UC15	70	70	LFV4	LFV5	D--4	F54K	F641	TW03	TW04	
F	FA07	FA08	AV53	ST05	ST06	ST06	LFV2	LFV3	D--3	F54L	TW02	TW01		
F	FA05	FA16	F34M	ZC13	ZC14	ZC14	LBV4	LBV4	D--1	F7A9	ZH40	ZH41		
A	A025	A026	A026	AV38	AV41	UC14	LBV3	LBV4	D--1	TD90	F546	Z140	Z141	
A	A024	A023	AV50	AV48	XG15	ZG15	LBV1	LBV2	C--5	F44B	TD73	Z142	Z142	
A	A022	A012	A1751	AV49	XG13	XG14	LDV4	LDV5	C--4	F44H	ST07	N205		
A	A010	A011	F4A1	F242	71	71	LDV2	LDV3	C--3	TD91	71	72		
A	A008	A008	AV15	AV13	72	71	LFV8	LDV1	C--2	F44G	ST03	S104		
A	A002	A003	F4A3	F34R	72	72	GF38	GF39	C--1	F44H	FTD0	ST01	ST02	
			AV40	AV40			GF30	GF37	FY3	FEAM	TD95			
			AV39	AV37			L1V4	L1V5	FY2	F44D	F646			
			AV54	AV52			L1V2	L1V3	FY1	TD76	F76			
			F248	FS41			L1V1	L2V5	FV3	F447	F76			
			A2AF	F307			L2V3	L2V4	FV2	F444	F642			
			TD95	TD95			L2V1	L2V2	FV1	F441	TD72			
									E--2					
									E..1					
									B--3					
									B--2					
									B..1					
									A..5					
									A..3					
									A..2					
									A..1					
									A..4					
Row		8	9	10	11	12	13							
		SS-J	SS-J	SS-3	SS-3	BB7	SS-J							
G	GB38	GB39	AI19	AA47	FK08	TA03	TD16	H..5	T055	TD40				
G	GA30	GA30	AI97	AI17	FK06	FK07	TD36	TD25	H..4	T056	TD44			
L	LHV1	LHV4	AI95	AI95	FK04	FK05	F84G	TA00	H..3	T052	TD45			
L	LHV6	YD98	AI94	AI96	FK02	FK03	TD10	TD93	H..2	T053	TD46			
G	GD30	YD99	AI91	AI92	FK00	FK01	TD13	F647	H..1	T054	TD43			
A	AI41	YD94	AI90	AI94	TH04	TH05	TD30	F..3	TD41	TD43				
A	AI39	YD10	AA46	AI79	TH02	TH03	TD23	F..3	T056	TD42				
A	AI37	YD98	AI16	AI61	TJ01	TJ02	F84R	TD33	F..2	TD70	TD41			
A	AI36	YD95	AI20	AI40	N203	N204	TA05	TD32	G..5	TD33	TD41			
A	AI40	YD03	AI19	AI77	N201	N202	TA01	TD11	G..4	TD36	TD41			
A	AI38	YD07	AI43	AI48			TD21	TA06	G..3	TD62	TD41			
G	GD39	YD91	AI93	AI42			TD15	TA02	G..2	TD66	TD41			
G	GD37	YD02	AI18	AA48			F84H	TD26	G..1	T055	TD44			
L	LHV2	LHV3	AI98	AI98			TD12	TD24	J..5	TD46	TD44			
G	GD38	GA37	AI78	AI15			TD22	TD14	J..4	TD42	TD46			
G	GD37	GD39	AI14	AI13			TD28	TD31	J..3	TD60	TD34			
								J..2						
								J..1						
									F..4					
									E..3					
									F..5					
									I..5					
									I..4					
									I..3					
									I..2					
									I..1					

they are loaded in the capsules. As with the previous tables, specimens loaded side by side appear in adjacent columns under the same row number. Table 7 shows the loading for the outer holder of 11J. In cases where more than one type of specimen is loaded into one slot, all types are listed at the column head. The loadings for the inner and outer central holders for 12J appear in Tables 8 and 9, respectively.

As can be seen in Tables 7 and 9, the TEM specimens are in the outer holders of each capsule, all in one hole in each capsule. The TEM specimens are loaded in tubes with welded end caps and placed into the holes provided. The tubes are of various lengths up to 25 mm in length. Most of the tubes are aluminum with slots for ease of disassembly, but some are zirconium and are completely sealed for protection of vanadium alloys. The loading of the tubes is shown in Tables 10 and 11 for 11J and 12J, respectively. The Zr tubes are indicated below the tube designation. Tubes 1-1 and 2-1 contain radioactive specimens from a previous irradiation in the FFTF. The specimens are listed in the order loaded so that the bottom specimen is listed first. Multiple columns are used simply to accommodate the large number of specimens; all are loaded one on top of the other.

Thermal Neutron Shield

Development of the thermal neutron shield presented the most difficult challenge of the irradiation experiment. The first task was to select the absorber material for the shield. Most available thermal absorbers can be excluded. Hafnium has been used as a thermal neutron shield in flux tailoring experiments in the HFIR,¹ but in the space available, only a factor of ten attenuation can be achieved. Gadolinium and cadmium have such high cross sections that they burn too fast to achieve the required exposure. Boron can be made to last for the duration of the experiment, in this case about one year, especially since it may be enriched in the ¹⁰B isotope. However, since the absorption reactions are (n, α), helium will collect in the shield container and produce a gas pressure of about 70 MPa, 10,000 psi, in the duration of the irradiation. This would necessitate using precious space for thick container walls. Europium can be used because the two isotopes initially present transmute into five additional isotopes of Eu and five isotopes of Gd, many of which have cross sections greater than 1000 barns. Thus each initial atom of Eu can ultimately absorb several neutrons.

Having chosen Eu for the absorber, many restrictions involving practicality and reactor safety must be considered. Europium is a very reactive metal necessitating glove box handling. Because of the reactivity and the fact that the atomic density of monoclinic Eu₂O₃ is 30% higher than Eu metal, europium oxide was selected as the absorber material. Europium oxide reacts with water to form a hydroxide which causes disintegration of the physical structure and swelling of about a factor of two. In the case of a leak in the can containing the shield, Eu₂O₃ could escape, and the can could swell and prevent removal from the reactor.

For the first line of defense, a shield can was designed such that two independent welds would have to fail in order to produce a leak. The design of the welds is illustrated in Fig. 3. In the event of a leak, a packed powder of Eu₂O₃ (which could easily be packed to 50% of theoretical density) would allow percolation of water and swelling in a matter of a few days at 70°C. To provide a second line of defense, the Eu₂O₃ was sintered to 92% of theoretical density to reduce percolation of water. In addition, the Eu₂O₃ was coated with aluminum to provide a water resistant coating.

A shield 413 mm in length was required to cover the specimen region of the capsule, but in order to reduce the shield to manageable sections, rings 25 to 50 mm in length were fabricated. The thickness of the rings was 1.85 mm and the outside diameter was 48.3 mm. A step 0.76 mm in length and of half the wall thickness was machined on each end in order to avoid neutron streaming through the cracks between the rings when stacked together. Several completed rings are shown in Fig. 4.

Table 7. Specimen Loading for the Central Outer Holder for HFIR-MFE-RB-11J

Center Section	Outer Holder	CVN	CVN	BB1, BB2, BB3, BB4	CVN	CVN	CVN	1.5 CVN
Row	1	2		3				6
Specimen Type								
A001	N201	Y605	Y606	DY31	DY32	DY13	TW05	L1V1
A002	N202	DY07	DY26	DY29	DY50	DY14	TW04	L1V3
A003	N203	DY08	DY25				FK03	L1V5
A004	N204	DY02	DY91				FK04	L2V1
A005	FA42	DY09	DY21	DY22	DY15		A021	L2V3
A006	FA49	D101	DT04				A023	TJ15
FA38	FA50	Y601	Y607				A024	TJ14
FA39	FA51	D138	DY40				A025	TJ13
FA40	FA54	DY01	DY05				A026	TJ12
FA41	FA55	DY37	DY39					LFV7
		Y602	Y604					LFV9
		D103	DT06	DY24	DY16			LBV1
		DY10	DY23					LBV3
		DY05	DY12	DY33	DY34	DY17		LBV4
		DY11	DY27					LBV5
		DY03	DY18	DY19				LBV6
		Y603	Y608					
Row	7	8	9	10	11	A	B	C
CVN	CVN	1.5 CVN	BB8	BB8	Pressurized Tubes	Pressurized Tubes	TEM Holders	
FK34	QC64	LHV1	LHV2	917 T4	919 M2	LFVG	L8VF	1-2
FK33	N205	LHV3	LHV4	1-6 SIC	13-18 SIC	LFVF	LBVD	1-1
FK32	FK01	LHV5	LHV6	T13	942 T4	LFVD	LBVB	1-8
FK31	FK00	LHV7	LHV8	917 M4	19-24 SIC	LFVB	LBVA	1-9
FK30	145Z1	LHV5	LHV9	843 M2	942 M4	LFVA	L2VF	1-10
	130Z1	144Z1	GF31	T11	917 T3	L2VA	L2VD	1-11
	129Z1	143Z1	GF33	843 T4	919 M1	LHVG	L2VB	1-12
	127Z1	142Z1	GF35	T7	943 M3	LHVF	L1VF	1-6
	126Z1	138Z1	LDV1	GF37	7-12 SIC	LHVD	L1VD	1-5
	125Z1	QC63	LDV2	843 M1	919 T2	LHVB	L1VB	1-4
		LDV4	LDV5			LHVA	L1VA	
		LDV6	LDV7					
		LDV8	LDV9					

Table 8. Specimen Loading for the Central Inner Holder for the HFIR-MFE-RB-12J

Center Section	Inner Holder								
Row	1 SS-J	2 SS-J	3 SS-J	4 SS-J	5 1.5 GVN				
TH61	TH56	F7F0	F6A8	AA87	AF17	AFL2	LFW6		
TH46	TH74	F8AL	F7A5	AF72	AA90	AF11	AFK5		
TH73	TH71	F6AL	F5A4	AA97	AF18	AF13	AFL8	LFW4	
TH58	TH63	F5AG	F4AF	AFK9	AA95	AFK3	AFL9	LFW3	
TH51	TH55	TB06	TB05	AA99	AA92	LDW2	AFL6	LFW2	
TH77	TH75	F4AM	F6A9	AF70	AFK1	LFW1			
TH66	TH72	TB00	TB03	AA94	AA98	LBW2	LHW2		
TH80	TH64	TB07	TB04	AF75	AA96	L1W2	L2W1	L1W6	
TH53	TH62	F7AF	F8AM	AFJ0	AA89	LHW1	LFW3	L1W6	
TH47	TH54	F6AS	F7AA	AF71	AF67	L1W1	LBW1	L1W4	
TH48	TH76	F7G0	F6AD	AF76	AF73	LFW2	LDW1	L1W3	
TH60	TH57	F8AB	F8AD	AA91	AA88	AFK7	AFK8	L1W2	
TH50	TH70	F8A4	F7A2	AF74	AFJ9	AFM2	AFM1	L1W1	
TH81	TH65	TB01	TB02	AF69	AA93	AFL4	AFK6		
TH67	TH52	F6AK	F8A9	AF68	AA86	AFL5	AFK4		
TH45	TH78	AV99	AV98	AFK0	AFK2	AFL0	TH38		
Row	6 BB5, BB7	7 SS-3	8 BB7	9 SS-3	BB7	BB7	BB7	BB7	BB7
FY6	N206	N207	G++5	71	71	H++3	TH06	TH07	
FY5	FA14	FA15	C++3	70	70	A++5	TW07	TW08	
FY4	A056	A055	I+-3	UC16	UC17	E++4	TJ05	TJ06	
FX7	A038	A039	I++4	ZC16	ZC17	D++2	FK18	FK19	
FX6	A036	FA13	H++5	ST10	ST11	M++2	FK16	FK17	
FX5	A034	A035	D++4	ST08	ST09	B--4	FK14	FK15	
FX4	A031	A033	H++4	XC17	XC16	C++2	FK12	FK13	
FX3	A029	A030	G++2	TJ07	TJ08	A++2	FK10	FK11	
FX2	FA16	FA17	F++1	Z144	Z145	J++1	TW05	TW06	
FX1	N208	N209	F++5	Z146	Z147	D++5	TH08	TH09	
G++1			A++3			B++4			
F++1			H++2			J++2			
E++1			C++4			E--4			
D++1			F++2			B++3			
C++1			C++5			K++1			
B++1			H++1			E--5			
A++1			D++3			B--5			
FZ7			E++5			I++2			
FZ6			J++5			G++3			
FZ5			J++4			M++1			
FZ4			G++4			K++2			
FZ3			J++3			K++2			
FZ2			E++3			K++3			
FZ1			A++4			E++2			
FVA			I++1			B++2			
FVD			F++4			B++5			

Table 9. Specimen Loading for the Central Outer Holder for the HEB-MEE-RB-12.

Table 10. Loading of TEM Specimens In HFIR-MFE-RB-11J

Holder	1-1		1-2		1-4		1-5	
				(Zr)				
Bottom	G604	KKG1	TE10	TE70	TED0	F2AG	F4D5	F65L
Y655	G603	KKG2	TE11	TE71	TED1	F2AF	F4D8	F65A
YS73	G602	KKG3	TE12	TE72	TED2	TEJ0	TEK3	F65F
YY52	G610	KKB2	TE13	TE73	TED3	F2AM	F4DR	F65H
YY55	G609	KKH1	TE14	TE74	TED4	F2AS	F4DG	F659
YY72	G612	KKH2	TE20	TE80	TE0	TEJ1	TEK4	F657
YZ55	G611	YD59	TE21	TE81	TE1	F2A8	F4DD	TEM0
YZ73	G601	YB38	TE22	TE82	TE2	F2AK	D4DF	TEM1
N234	KK1	KK11	TE23	TE83	TE3	TEJ2	TEL0	TEM2
N235	KK2	KK12	TE24	TE84	TE4	F2AA	F56F	TEM3
N236	KK3	KK13	TE30	TE90	TE0	F2AH	F56S	F755
	KKB3	KKJ1	TE31	TE91	TEG1	TEJ3	TEL1	F75V
	KKC1	KKJ2	TE32	TE92	TEG2	F374	F565	F75D
	KKC2	KKJ3	TE33	TE93	TEG3	F37X	F56A	F75G
	KKC3	JAR1	TE34	TE94	TEG4	TEJ4	TEL2	F75H
	KKD1	TW36	TE40	TE00	TEH0	F378	F56G	F75S
	KKD2		TE41	TE01	TEH1	F37K	F56D	F75L
	KKD3		TE42	TE02	TEH2	TEK0	TEL3	F754
	KKE2		TE43	TE03	TEH3	F37M	F65R	F868
	KKF1		TE44	TE04	TEH4	F37F	F655	F86T
	KKF2		TE50	TEA0	TEK1	TEL4	F86V	
	KKF3		TE51	TEA1	F4D9		F86H	
			TE52	TEA2	F4DB		F866	
			TE53	TEA3	TEK2		F86L	
			TE54	TEA4				
			TE60	TEB1				
			TE61	TEB2				
			TE62	TEB3				
			TE63	TEB4				
			TE64					

Table 10. Loading of TEM Specimens in HFIR-MFE-RB-11J (cont'd)

Holder	1-6			1-8			1-9		
	(Zr)		(Zr)	GA3B	FML1	YB19	YB58	YB91	
Bottom	AV01	AV16	ND16	46	LFV1	GA3D	FML2	YB20	YB59
	AV02	AV17	ND10	XD25	LFV2	GA3F	FML3	YB21	YB93
	AV03	AV18	ND18	XD50	LFV3	GB3A	FR01	YB22	YB93
	AV04	AV19	ND05	XD45	LFV4	GB3B	FR02	YB23	YB94
	AV05	AV20	ND19	ZD26	LFV5	GB3D	FR03	YB24	YB95
	AV06	AV21	ND25	ZD50	LFV6	GB3F	FR04	YB25	A028
	AV07	AV22	ZD32	ZD30	LFV7	GG3D	YB01	YB26	A029
	AV08	AV23	ZD48	UD38	LFV8	GD3A	YB02	YB27	
	AV09	AV24	ZD47	UD45	LFV9	GD3B	YB03	YB28	
	AV10	AV25	ZD34	UD25	LFV/A	GF3B	YB04	YB29	
	AV11	AV26	ZD42	ND08	LFVB	GF3A	YB05	YB30	
	AV12	AV27	ZD36	ND20	LFVD	GF3B	YB06	YB31	
	AV13	AV28	ZD27	ND27	LFVF	GF3D	YB07	YB32	
	AV14	AV29	ZD34	ND09	LFVG	GF3F	YB08	YB33	
	AV15	AV30	UD33	ND02	LFVH	G03A	YB09	YB34	
	AV31	UD30	UD48	LBV1	G03B	YB10	YB35	YB73	
	AV32	UD46	ZD31	LBV2	G030	YB11	YB36	YB74	
	AV33	UD33	ZD38	LBV3	G03F	YB12	YB37	YB75	
	AV34	71	XD29	LBV4	G13A	YB13	YB38	YB76	
	AV35	71	XD43	LBV5	G13B	YB14	YB39	YB77	
	AV36	71	ND29	LBV6	G13D	YB15	YB40	YB78	
				L1V1	G13F	YB16	YB41	YB79	
				ND28	L1V2	FMA1	YB42	YB80	
				70	UD21	YB17	YB43	YB81	
				70	L1V3	FMA2	YB18	YB44	
				72	L1V4	FMA3	AB18	YB45	
				72	L1V5	FMD1	A025	YB46	
					L1V6	FMD2	YB47	YB47	
					L2V1	FMD3	YB48	YB48	
					L2V2	FMF1	YB49	YB49	
					L2V3	FMF2	YB50	YB50	
					L2V4	FMF3	YB51	YB51	
					L2V5	FMH1	YB52	YB52	
					L2V6	FMH2	YB53	YB53	
					LHV1	FMH3	YB54	YB54	
					LHV2	FMK1	YB55	YB55	
					LHV3	FMK2	YB56	YB56	
					LHV4	FMK3	YB57	YB57	
					LHV7				
					LHV8				
					LHV9				
					LHVA				

Table 10. Loading of TEM Specimens In HFIR-MFE-RB-11J (cont'd)

Table 10. Loading of TEM Specimens In HFIR-MFE-RB-11J (cont'd)							
Holder	1-10			1-11			1-12
Bottom	YB97	XR1T	AF13	AF46	AA07	LDV1	A001
	YB98	XR1X	AF14	AF47	AA08	LDV2	A002
	YB99	XR1Y	AF15	AF48	AA09	LDV3	A003
	YD50	XR21	AF16	AF49	AA10	LDV4	A004
	YD51	XR22	AF17	AF50	AA11	LDV5	A005
	YD52	XR23	AF18	AH51	AA12	LDV6	A006
	YD53	XR24	AF19	A030	AL01		A007
	YD54	XR25	AF20	A031	AL02		A008
	YD55	AA01	AF21	A032	AL03		A009
	YD56	AA02	AF22	TW25	AL04		A010
	YD57	AA03	AF23	CAPA?	AL05		FA14
	YD58	AA04	AF24	N232	AL06		FA19
	YD60	AA05	AF25		AL07		FA20
	YD61	AA06	AF26		AL08		FA10
	XR11	AF01	AF27		AL09		FA31
	XR12	AF02	AF38		AL10		FA15
	XR13	AF03	AF29		AL11		FA12
	XR14	AF04	AF30		AL12		N201
	XR15	AF05	AF31		AC01		XH63
	XR16	AF06	AF32		AC02		HX68
	XR17	AF07	AF33		AC03		XH69
	XR18	AF08	AF34		AC04		N202
	XR19	AF09	AF35		AC05		N203
	XR1A	AF10	AF36		AC06		N204
	XR1B	AF11	AF37		B000		N205
	XR1D	AF12	AF38		B001		N206
	XR1F		AF39		B100		N207
	XR1G		AF40		B101		N208
	XR1H		AF41		B200		N209
	XR1K		AF42		B201		N210
	XR1L		AF43		B300		
	XR1M		AF44		B301		
	XR1R		AF45				
	XR1S						

Table 11. Loading of TEM Specimens In HFIR-MFE-RB-12J

Holder	2-1		2-2		2-4		2-5	
					(Zr)			(Zr)
Bottom	YS53	G605	KKE-1	TE15	TE75	TED5	F2AB	FD4A
	YS54	G606	KKE-2	TE16	TE76	TED6	F2AB	F4DS
	YS56	G607	KKF-1	TE17	TE77	TED7	TEJ5	TEK7
	YS57	G608	KKF-2	TE18	TE78	TED8	F2AL	F4DW
	YY53	G613	KKF-3	TE19	TE79	TED9	F2A4	FRDZ
	YY54	G614	KKG-1	TE25	TE85	TEE5	TEJ6	TEK8
	YY56	G615	KKG-2	TE26	TE86	TEE6	F2AT	F4F1
	YY57	G616	KKG-3	TE27	TE87	TEE7	F2A7	F4F3
	YY74	YC42	KKH-1	TE28	TE88	TEE8	TEJ7	TEK9
	YY75	AFB9	KKH-2	TE29	TE89	TEE9	F2AD	F4F4
	YZ53	KKI-1	KKH-3	TE35	TE95	TEG5	F2AV	F4F5
	YZ54	KKI-2	KKI-1	TE36	TE96	TEG6	TEJ8	TEL5
	YZ56	KKI-3	KKI-2	TE37	TE97	TEG7	F379	F563
	YZ57	KKB-1	KKI-3	TE38	TE98	TEG8	F37A	F565
	YZ74	KKB-2	KKJ-1	TE39	TE99	TEG9	TEJ9	TEL6
	YZ75	KKC-1	KKJ-2	TE45	TE05	TEM5	F37G	F56B
	YS74	KKC-2		TE46	TE06	TEM6	F37H	F56H
	YS75	KKC-3		TE47	TE07	TEM7	TEK5	TEL7
		KKD-1		TE48	TE08	TEM8	F37W	F56M
		KKD-2		TE49	TE09	TEH9	F37Y	F56T
		KKD-3		TE55	TEA5		TEK6	TEL8
				TE56	TEA6			F65T
				TE57	TEA7			F654
				TE58	TEA8			F86M
				TE59	TEA9			TEL9
				TE65	TEB5			F86R
				TE66	TEB6			
				TE67	TEB7			
				TE68	TEB8			
				TE69	TEB9			

Table 11. Loading of TEM Specimens In HFIR-MFE-RB-121 (cont'd)

Holder	2-6			2-8			2-9		
	(Zr)			LHW1	G05A	FMB1	FR05	YC12	YC26
Bottom	AV66	ND15	71	LFW1	LHW2	G05B	FMB2	FR06	YC48
	AV67	ND14	71	LFW2	LHW3	G05D	FMB3	FR07	YC13
	AV68	ND30	46	LFW3	LHW4	G15A	FMB1	F409	YC49
	AV69	ND24	46	LFW4	LHW5	G15B	FMB2	FR0A	YC14
	AV70	ND03	72	LFW5	LHW6	G15B	FMB3	FR0D	YC50
	AV71	ND12	72	LFW6	LHW7	G15D	FMB4	FR0F	YC15
	AV72	XD22	71	LFW7	LHW8	G15F	FMAA	YC01	YC29
	AV73	XD26	71	LFW8	LHW9	FMA4	FMBB	YC02	YC39
	AV74	XD41	71	LFW9	LHW1A	FMA6	FMB8	YC03	YC30
	AV75	XD49	46	LFW1A	G45A	FMA7	FMDA	YC59	YC32
	AV76	ZD37	46	LFW1B	G45B	FMD4	FMDB	YC04	YC37
	AV77	ZD25		LFW1D	G45B	FMD5	FMDA	YC06	YC16
	AV78	ZD21		LFW1F	G45D	FMDG	FMBF	YC07	YC54
	AV79	ZD40		LFW1G	G45F	FMF4	FMDA	YC43	YC41
	AV80	UD43		LFW1H	G45A	FMB5	FMB8	YC08	YC35
	AV81	UD20		LBW1	GB5B	FMBD	FMHA	YC44	YC58
	AV82	UD40		LBW2	GB5D	FMBE	FMBH	YC09	YC38
	AV83	UD32		LBW3	GB5F	FMBF	FMK4	YC21	XR38
	AV84	71		LBW4	GG5A	FMB5	FMKB	YC10	YC37
	AV85	71		LBW5	GG5D	FMB6	FMLA	YC46	YC54
	AV86	71		LBW6	GD5A	FMK4	FMLB	YC11	YC36
	AV87	70		L1W1	GD5B	FMK5	FML6	YC47	XR39
	AV88	70		L1W2	GD5F	FMK6			
	AV89	70		L1W3	GF5A	FML4			
	AV90	72		L1W4	GF5B	FML5			
	AV91	72		L1W5	GF5D	FML6			
				L1W6	GF5F				
				L2W1					
				L2W2					
				L2W3					
				L2W4					
				L2W5					
				L2W6					

Table 11. Loading of TEM Specimens In HFIR-MFE-RB-12 (cont'd)

Holder	2-10	2-11	2-11	2-12
Bottom				
YC31	AFA8	AFD1	AFF1	AA55
YC65	YC78	YC92	YD76	XF2R
YC32	AFA9	AFD2	AFF2	AA56
YC66	YC79	YC93	YD77	XF2S
YC33	AFA0	AFD3	AFF3	AFG5
YC67	YC80	YC94	YD78	XF28
YC68	AFB1	AFD4	AFF4	AFG6
YC35	YC81	YC95	YD79	AFG7
YC69	AFB2	AFD5	AFF5	XF2A
YC36	YC82	YC96	YD80	AFG8
YC70	AFB3	AFD6	AFF6	AFG9
AFA1	YC83	Y097	YD81	XF2D
YC71	AFB4	AFD7	AFF7	AFG0
AFA2	YC84	YC98	YD82	XF2G
YC72	AFB5	AFD8	AFF8	AA51
AFA3	YC85	YC99	YD83	XF2H
YC73	AFB6	AFD9	AFF9	AA52
AFA4	YC86	AFD0	YD84	XF2K
YC74	AFB7	TW34	AFF0	AA53
AFA5	YC87	TW35	YD85	XF2L
YC75	AFB8	TW37	AFG1	AA54
AFA6	YC88	TW30	YD86	XF2M
YC76	YC89	TW44	N231	AFG2
AFA7	AFB0	TW40	N237	YD87
YC77	YC90	A037	N243	
		A038	N229	
		A043	N230	
		A027		
		A044		

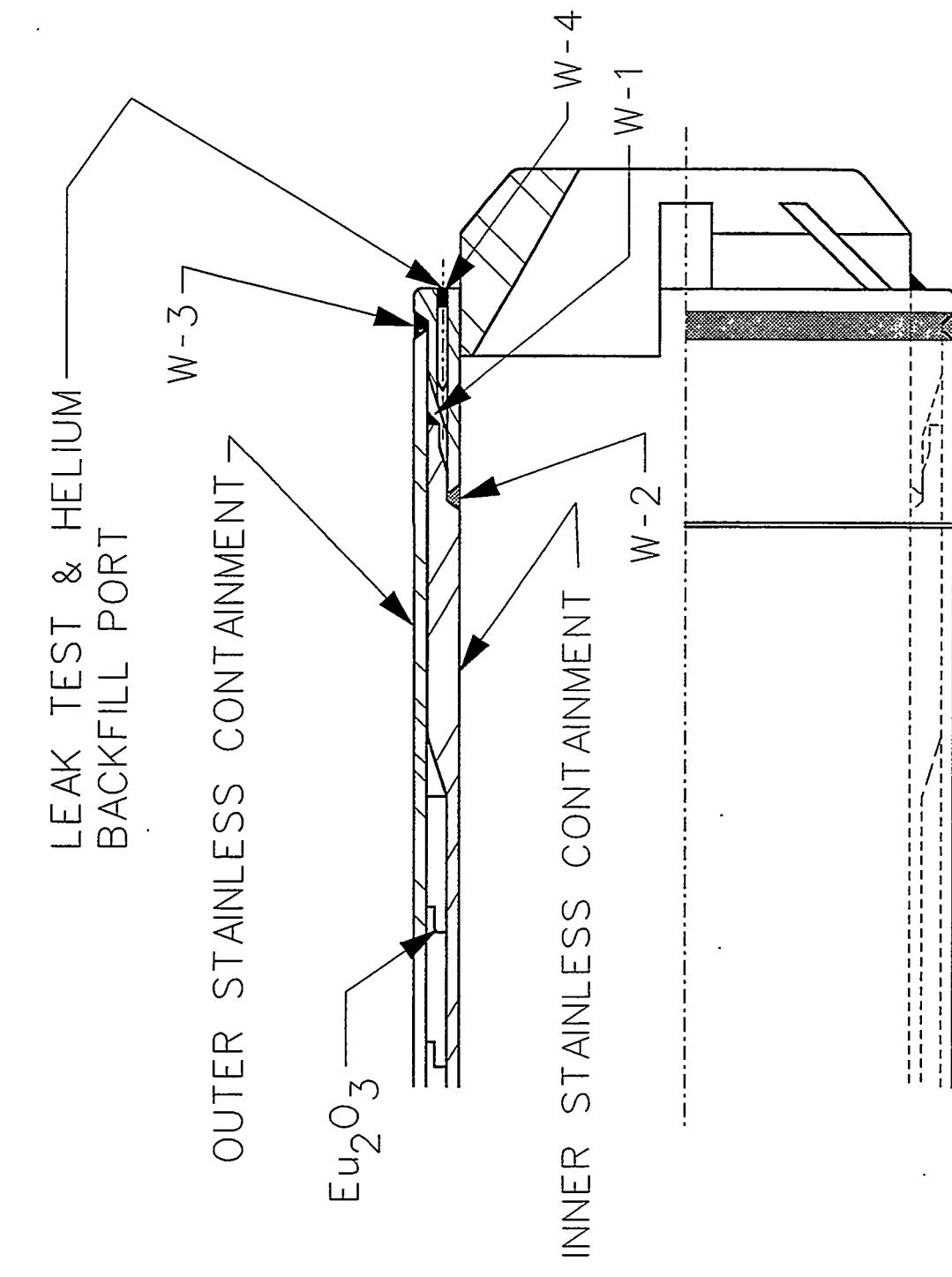


Fig. 3. Bottom section of the shield can showing the details of the seal welds. Weld W-1 and one of W-2, W-3, or W-4 must fail in order to produce a leak. W-4 is the seal for the helium back-fill port to provide a means of leak checking the welds.

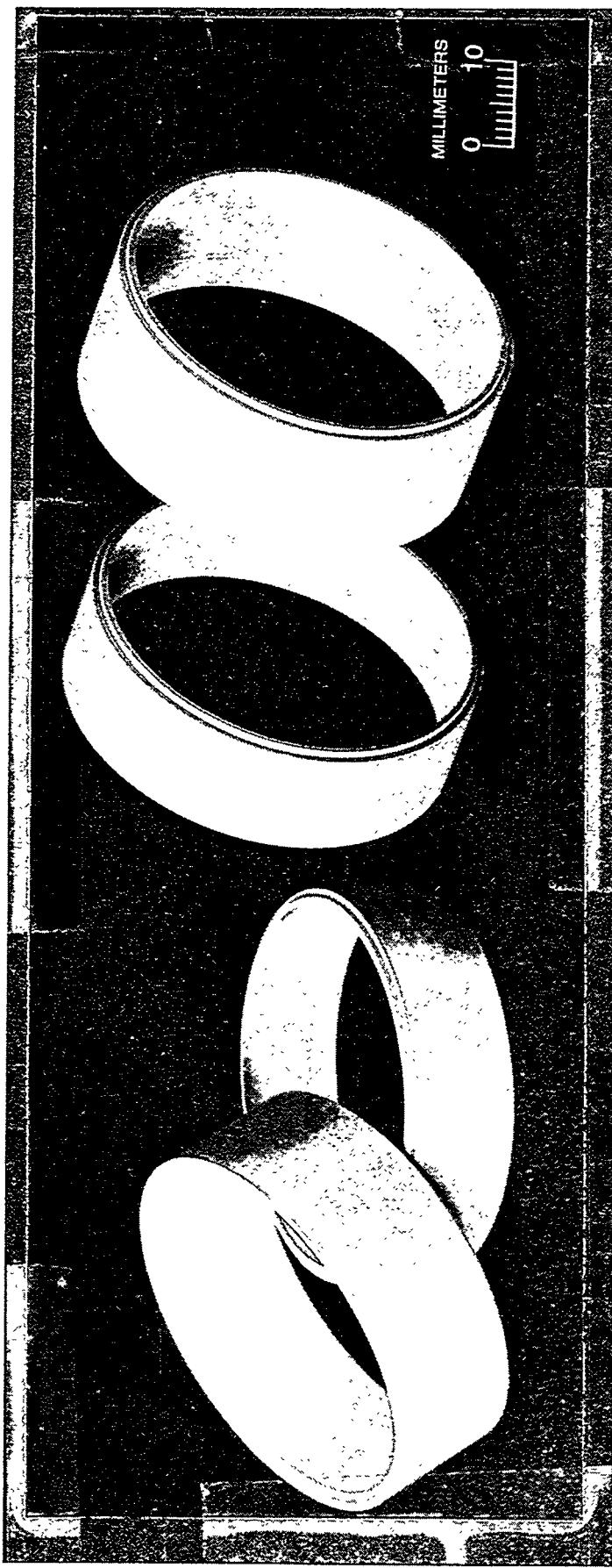


Fig. 4. Rings of europium oxide prior to coating with aluminum and assembling into a shield.

The rings were fabricated by cold pressing and sintering. Cubic Eu₂O₃ powder was isopressed and calcined at 1800°C to transform it to the more dense monoclinic phase. Following crushing, grinding, and milling operations, a binder was introduced to facilitate die pressing. The powder was then pressed in a steel die at 310 MPa, resulting in a compact of 65% of theoretical density. The binder was then burned out at 1300°C in an air atmosphere. The rings were then sintered at 1730°C for one hour in an argon-4%H atmosphere. The final shape was machined to a tolerance of 0.03 mm.* Coating with aluminum was done by plasma sputtering to produce a coating 2-4 µm in thickness. Since the coating produced by this technique was porous, it was sintered in a vacuum at 576°C, 0.9 of the melting point of aluminum, for 4 hours.

The rings were then fitted together on the inner wall of the shield can to form the cylindrical shield pictured in Fig. 5. The ends of the capsules were shielded with sections of a HFIR control plate containing Eu₂O₃ in an Al matrix. Space was not a constraint so that two such 25 mm plates were used and were placed in the specimen chamber which had a monitored containment. In the case of a leak, a high moisture level in the purge gas could be detected within a few minutes.

Thermal Performance

The temperature distribution in the capsules was examined carefully during the first cycle of operation. The temperature distributions are shown in Figs. 6 and 7 for 11J and 12J, respectively. The temperatures plotted are thermocouple temperatures. Specimen temperatures are higher according to heat conduction from the particular specimen and to local nuclear heating, which depends upon axial position and shielding. Calculated corrections to thermocouple temperatures are given in Figs. 6 and 7. It is apparent that the end regions are cooler than expected in both capsules. In 11J, the ends were about 50°C lower than the design temperature, and in 12J the end regions were about 150°C below the design temperature.

It is believed that the low temperatures are the result of two causes: end losses and centering pad losses. A few specimens in the outer middle region are also affected by centering pad losses. Centering pads are used to maintain the position of the specimen holder in the capsule and to maintain the gap needed for the flow of the control gas. However, the centering pads also provide a pathway for heat loss. The localized temperature depression is approximately 20°C (3%) in 11J and 50°C (6%) in 12J. The centering pads affect only those specimens that are near centering pad locations, along with all the upper and lower regions.

The majority of the specimens in both capsules is operating within the desired temperature bands: ±20 C for both the 300 and 500°C capsules. The average of the central thermocouples was chosen for the control temperature in both capsules. The end regions are considered to be at 250 and 350°C for 11J and 12J, respectively. As with any irradiation experiment that does not contain liquid metal, variations between specimens are expected.

Conclusions

Two unique first of a kind irradiation experiments have begun irradiation in the HFIR. The newly developed europium oxide thermal shields permit fast reactor irradiations in the HFIR including, if desired, irradiation at temperatures as low as 60°C. The experiments contain a very large number of specimens of alloys and ceramics of many specimen configurations.

The temperatures at the ends of the capsules were slightly lower than the design temperatures, but this is not unexpected in an experiment of such a new design. The low temperatures are attributed to undesirable heat conduction through centering pads. The design will be modified in future irradiation experiments to alleviate this condition. The experiment will serve as a prototype for other collaborative HFIR experiments between Japan and the U.S.

*Machining was performed by Chand Associated, Inc., Worcester, MA.

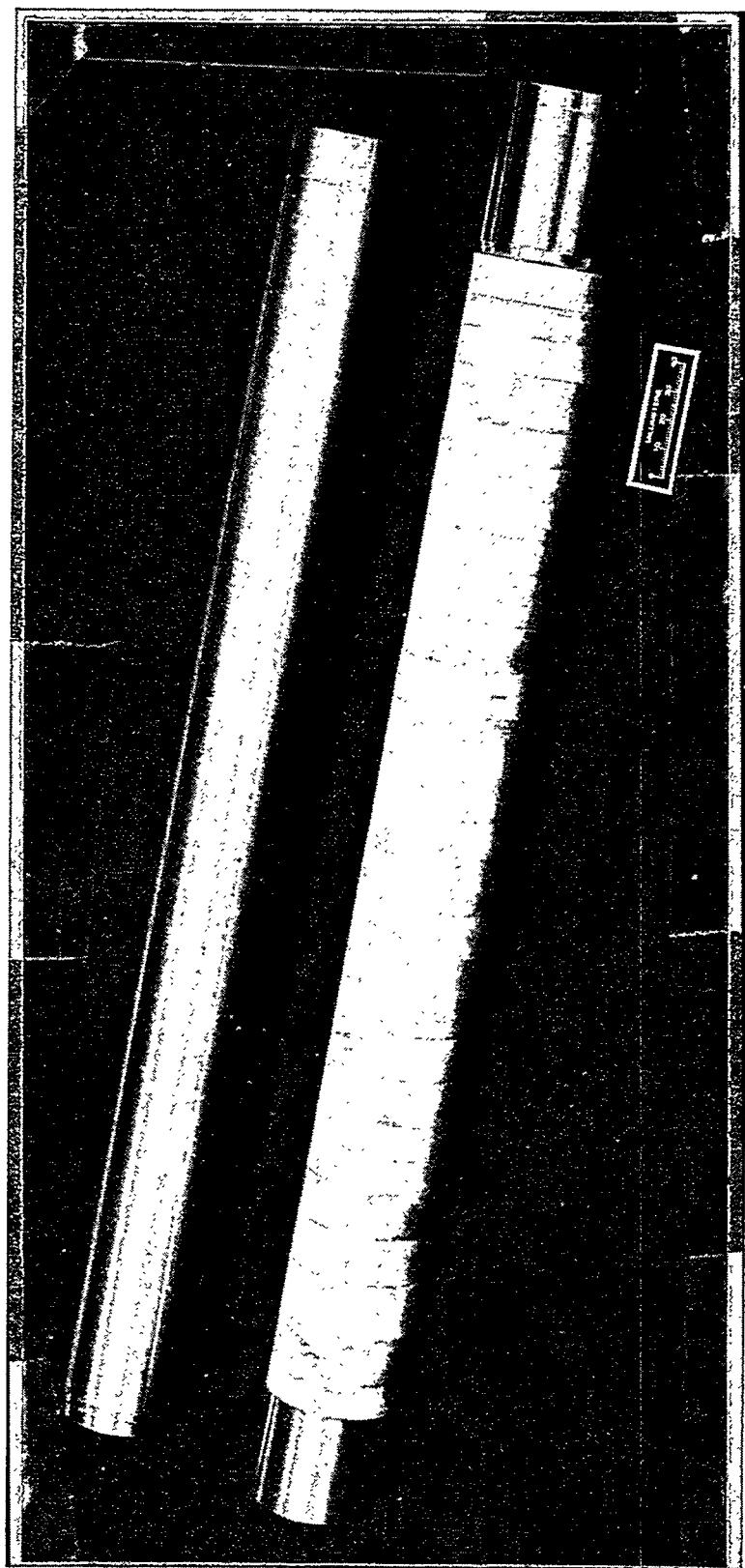


Fig. 5. Europium oxide rings in place on the inner wall of the shield can. The outer wall is at the top in the figure and will be fitted over the europium rings and welded in place.

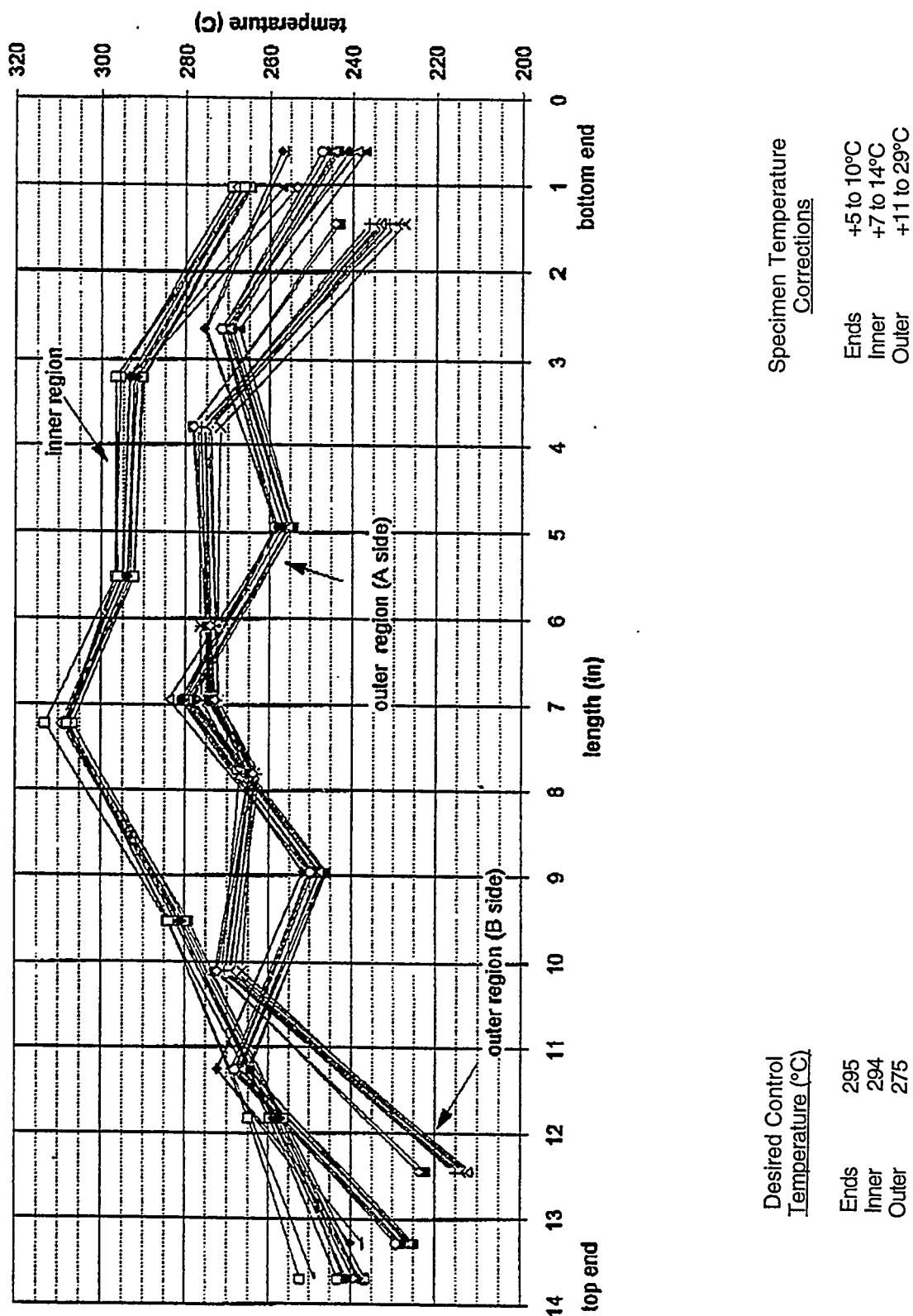


Fig. 6. Temperature data for the first cycle of operation of HFIR-RB-11J.

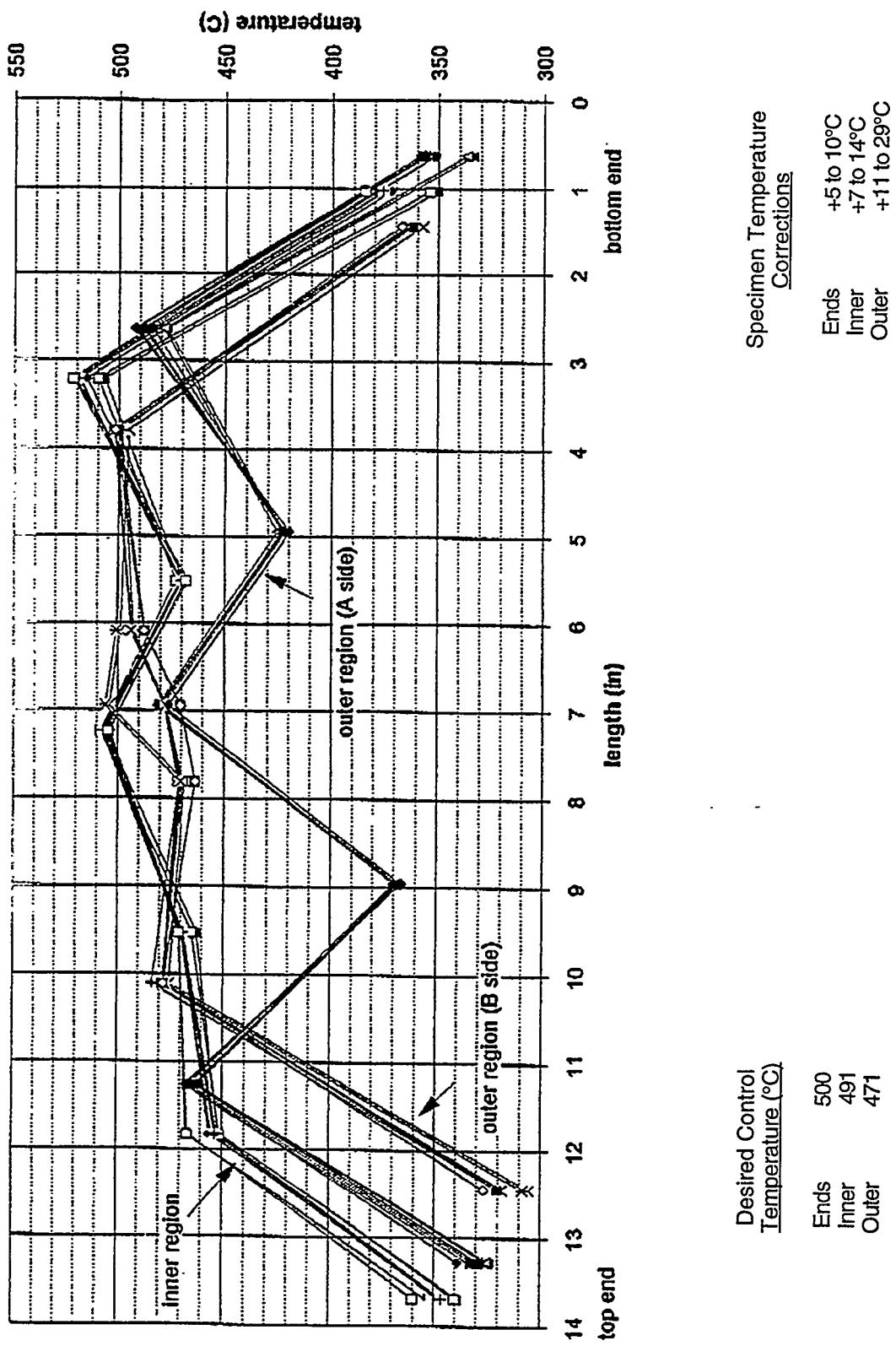


Fig. 7. Temperature data for the first cycle of operation of HFIR-RB-12J.

REFERENCES

1. A. W. Longest, J. E. Corum, D. W. Heatherly, and K. R. Thoms, "Design of Spectrally Tailored Fusion Reactor Materials Experiments in the HFIR RB* Capsule Irradiation Facility, *J. Nucl. Mater.* 155-157 (1988), 1346.