

NEUTRON IRRADIATION OF V-Cr-Ti ALLOYS IN THE BOR-60 FAST REACTOR: DESCRIPTION OF THE FUSION-1 EXPERIMENT — A. F. Rowcliffe (Oak Ridge National Laboratory), H. C. Tsai and D. L. Smith (Argonne National Laboratory), and V. Kazakov, and V. Chakin (RIAR, Dimitrovgrad)

SUMMARY

The FUSION-1 irradiation capsule was inserted in Row 5 of the BOR-60 fast reactor in June 1995. The capsule contains a collaborative RF/U.S. experiment to investigate the irradiation performance of V-Cr-Ti alloys in the temperature range 310 to 350°C. This report describes the capsule layout, specimen fabrication history, and the detailed test matrix for the U.S. specimens. A description of the operating history and neutronics will be presented in the next semiannual report.

INTRODUCTION

Low activation alloys based on the V-Cr-Ti system are the primary option for the first wall and blanket structure for liquid metal-cooled fusion reactor systems. A promising combination of fabricability, strength, ductility, and toughness has been identified within the composition range V-(4-5)Cr-(4-5)Ti. Initial results from fast reactor irradiation experiments conducted over the range 420-600°C indicated very little change in the ductile-to-brittle transition temperature (DBTT) for a V-4Cr-4Ti alloy irradiated to neutron doses in the range 24-36 dpa. However, large upward shifts in DBTT have been reported for a similar alloy irradiated at lower temperatures [1].

This paper describes the construction and operation of a collaborative RF/U.S. irradiation experiment in the BOR-60 reactor designed to investigate the irradiation performance of vanadium alloys in the temperature range 310-350°C for neutron doses in the range 15-20 dpa. The capsule design and specimen fabrication history are described, and details of the specimen test matrix are provided. The operating history, neutronics, and experiment disassembly will be provided in the next semiannual report.

CAPSULE CONSTRUCTION

The layout of the capsule is shown in Fig. 1. The experiment is contained inside a cylindrical tube measuring 41 mm ID × 1 mm thick, which is housed inside a hexagonal duct measuring 44 mm flat-to-flat. The space between the outer duct and inner tube is filled with argon to provide thermal insulation against neighboring fuel assemblies. The experimental capsule is a stainless steel cylindrical tube measuring 36 mm ID × 2 mm thick filled with ⁷Li of 99.9% purity. The outside of the capsule is in contact with flowing sodium. Both U.S. and RF specimens are supported on a set of 10 tiers with the U.S. specimens occupying the lower six, as shown in Fig. 2. Each of the U.S. tiers has an upper and a lower bulkhead. There are three holes at the periphery of the bulkheads and connecting rods through these three holes join the six tiers together to form an assembly. The connecting rods are welded to the bulkheads at the top and bottom ends and the assembly placed over the bottom end plug of the capsule.

Within each US. tier, the tensile (SS-3), Charpy (CVN), and disk compact tension (DCT) specimens are separated from each other by coil-shaped 0.4-0.5 mm dia molybdenum wires. The TEM disks are contained in perforated stainless steel tubes. The SS-3 and CVN specimens are held upright and bundled into groups with 0.2 mm dia nichrome wires. The DCTs are stacked and aligned with free-standing stainless steel rods through the pull holes. All specimens are tied with nichrome wires around the three connecting rods to form a bundle; this arrangement prevents the specimens from slipping off the edge of the bulkheads.

After loading the specimens, the capsule was shipped to the Institute of Physics and Power Engineering at Obninsk for Li-filling and final weld closure. The ⁷Li was first purified by distillation and Zr-gettering using the equipment shown in Fig. 3. This process lowered the nitrogen content

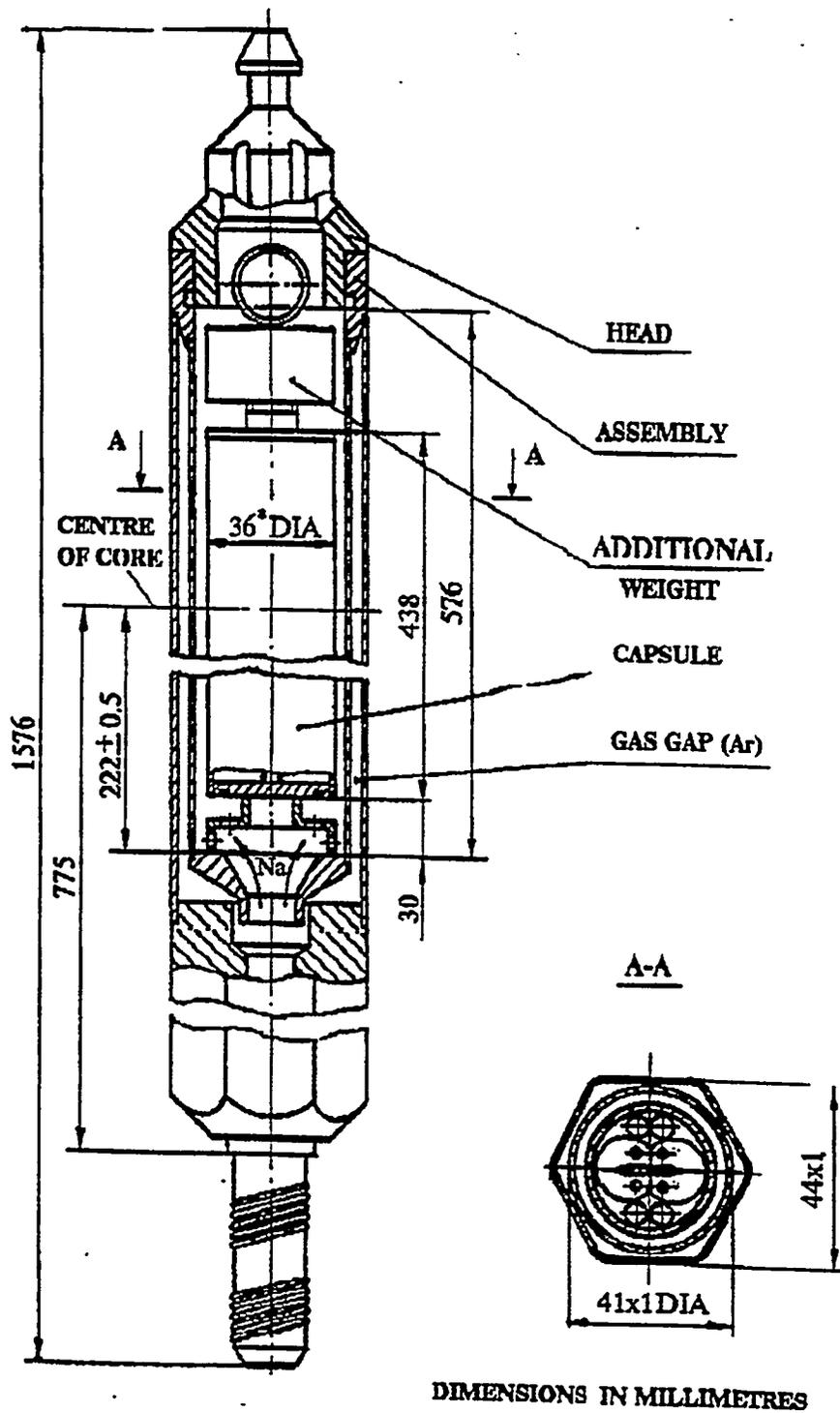


Fig. 1. Schematic of the Fusion-1 Capsule for BOR-60.

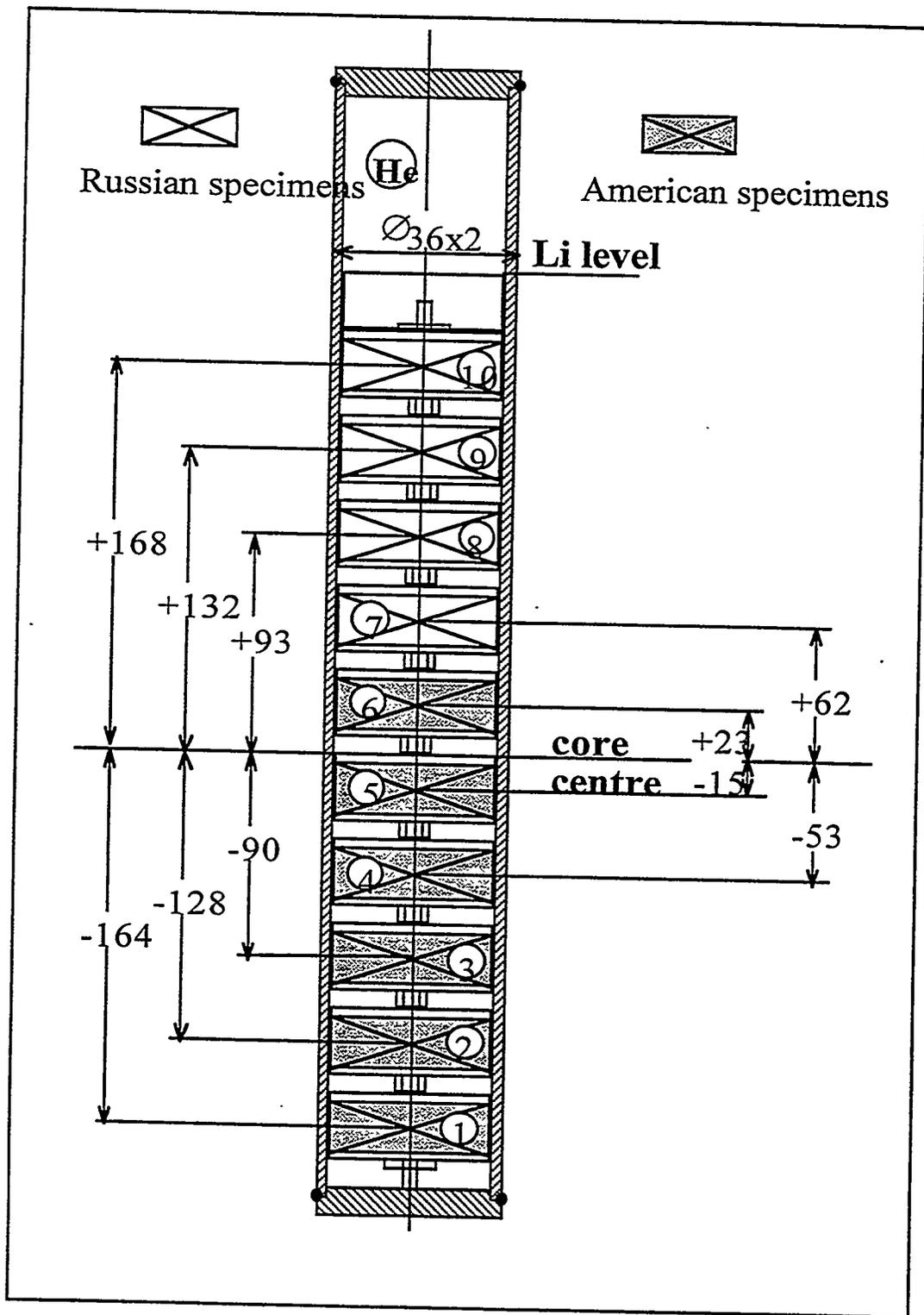


Fig. 2. Distribution of U.S. and RF Specimens within the Fusion-1 Capsule.

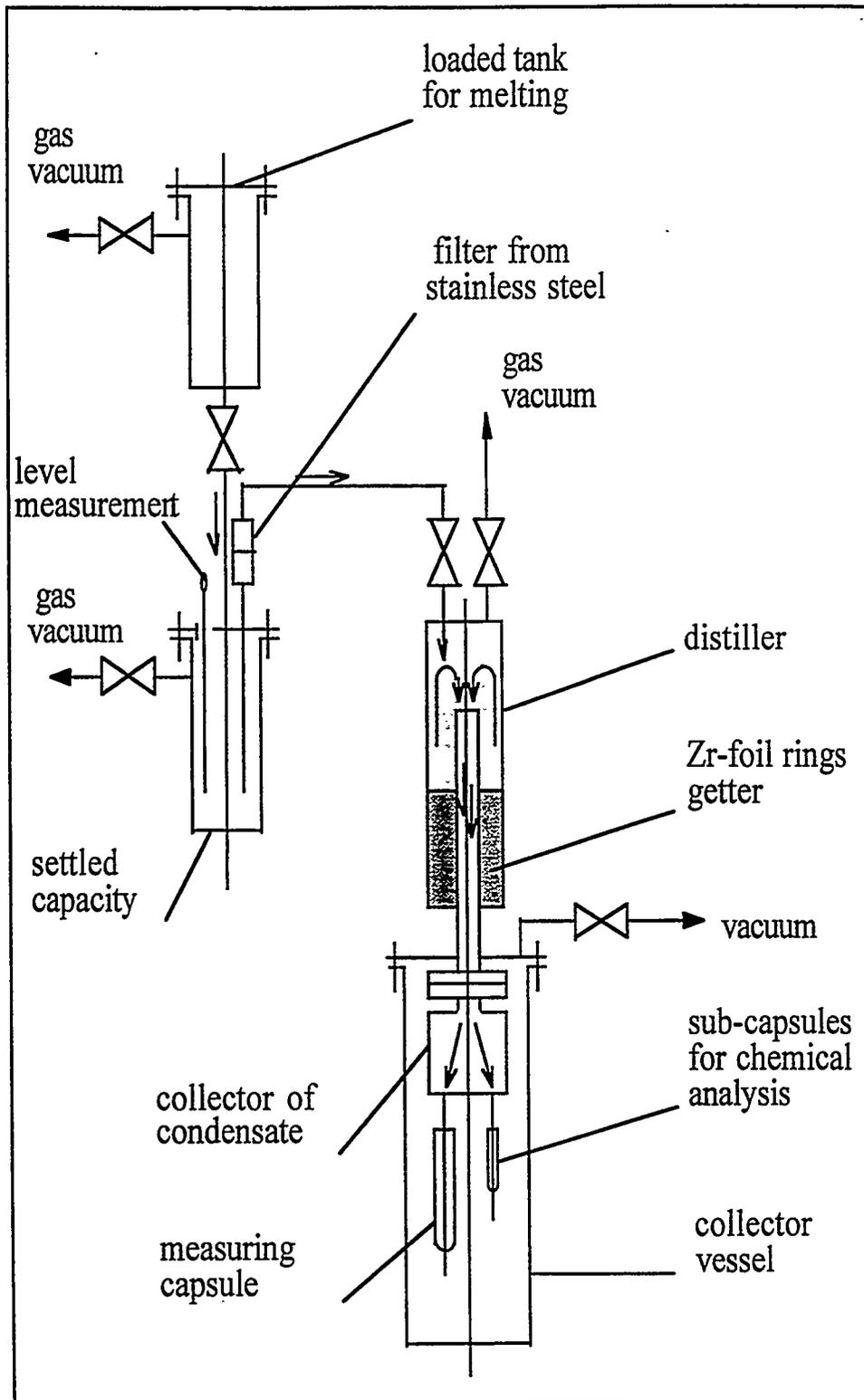


Fig. 3. Li Distillation and Purification System

from 200 to <3 wppm and reduced the carbon content to ~30 wppm. To reduce the probability of forming cavities, the capsule was mechanically vibrated during Li-filling at 250-300°C using the arrangement shown in Fig. 4. Vibration was continued for about 15 min and the vacuum was maintained at better than 10^{-4} torr. Following x-radiography, the capsule was shipped back to RIAR, assembled, and inserted in the G-23 Cell of Row 5 of the BOR-60 reactor on June 27, 1995.

U.S. ALLOYS AND SPECIMEN FABRICATION HISTORY

The primary focus of the U.S. experiment was on the fracture behavior of the 500 kg heat of V-4Cr-4Ti, heat no. 832665. In addition, specimens were also prepared from eight experimental heats with small variations in Cr and Ti concentrations. The chemical analyses of these alloys are presented in Tables 1 and 2. The loading list and specimen identification codes are presented in Tables 3 and 4.

The fabrication histories of the various types of plate supplied by Teledyne Wah Chang, Albany for the 500 kg heat of V-4Cr-4Ti and for the 30 kg heat of compositional variants are summarized in Tables 5 and 6, respectively. Details of the fabrication history of the various sets of specimens are presented in Tables 7, 8, 9, and 10.

FUTURE WORK

The Fusion-1 capsule was successfully disassembled at RIAR and the specimens will be shipped back to the U.S. for postirradiation testing. A report on the operating history and neutronics will be presented in the next semiannual report.

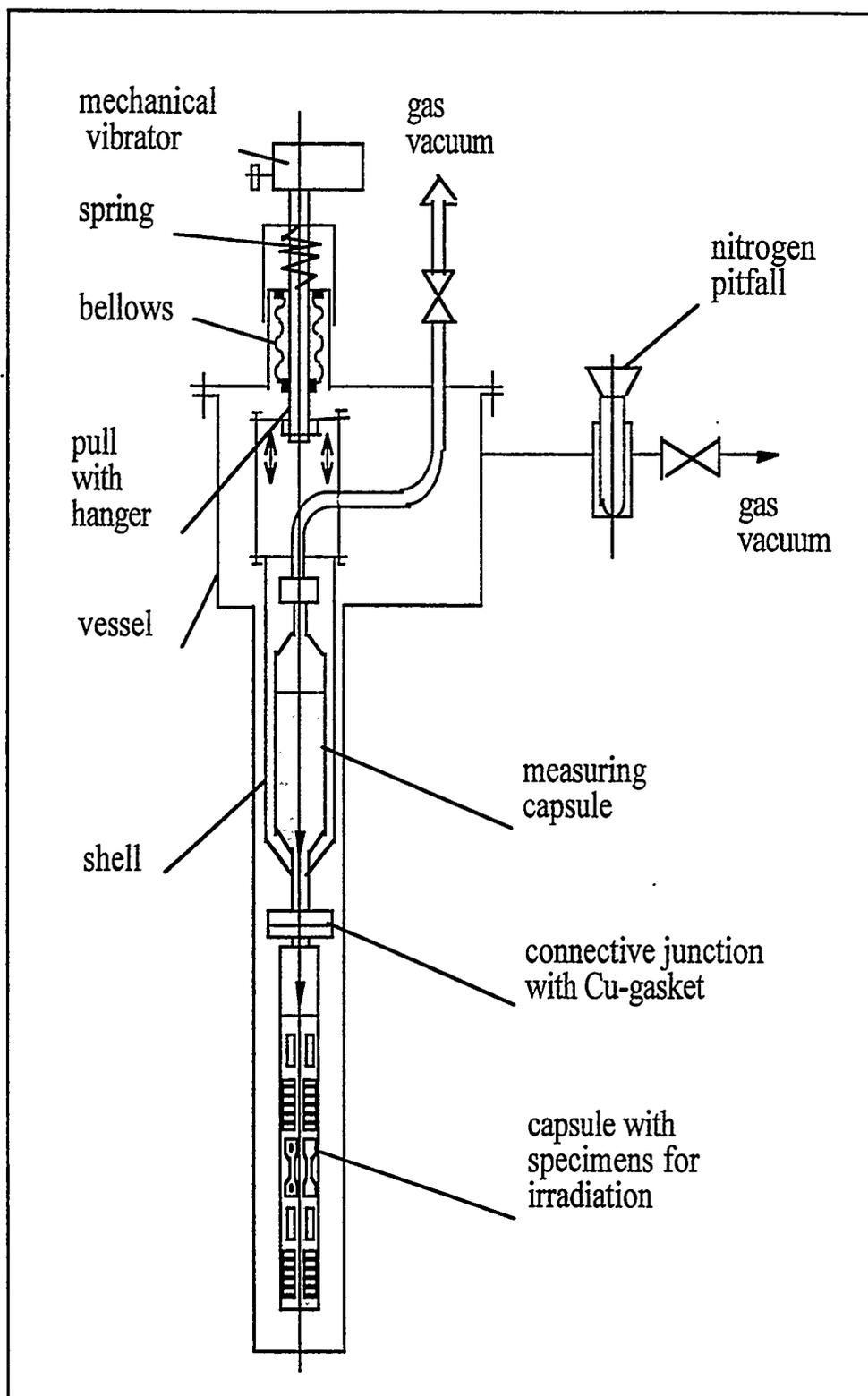


Fig. 4. System for Filling Capsule with Liquid Lithium.

Table 1. Chemical Analyses of 500 kg Heat (832665) of V-4C4-4Ti (wt. ppm)

| Element | GDMS Evans | GDMS Evans | GDMS ORNL | ICP-Emission ORNL | Ingot Wah Chang |
|---------|---------------|---------------|--------------|----------------------|--------------------|
| Ag | 0.11 | 0.045 | | | |
| Al | 450 | 260 | <200 | | 185 |
| As | <1.5 | <1.3 | <3 | | |
| Au | <.059 | <.065 | <6 int | | 7 |
| B | 4.1 | 3.3 | | | |
| Ba | <.23 | <.29 | | | |
| Be | <0.021 | <.0017 | | | |
| Bi | <.0075 | <.0061 | | | |
| Br | <.14 | <.092 | | | |
| C | <260 | <140 | <310 | | 86 |
| Ca | <.32 | <.20 | | | |
| Cd | <.19 | <.13 | | | |
| Ce | 0.017 | 0.021 | | | |
| Cl | <.087 | <.050 | | | <2 |
| Co | 0.31 | 0.28 | | | |
| Cr | %3.0 | 3.5 | %3.0 | %3.1 | %3.77 |
| Cs | <.024 | <.013 | | | <10 |
| Cu | 0.8 | 0.88 | | | <50 |
| F | <.081 | <0.66 | | | |
| Fe | 210 | 200 | 256 | | 220 |
| Ga | 1.3 | 1.5 | | | |
| Ge | <.030 | <.030 | | | |
| Hf | 8.4 | 8.3 | 10.8 | | |
| Hg | <.045 | <.025 | | | |
| I | <0.18 | <.015 | | | |
| In | 0.075 | 0.057 | | | |
| Ir | <.0030 | <.0032 | | | |
| K | <.051 | <.021 | | | |
| La | <.00079 | <.00064 | | | |
| Li | <.0017 | <.0014 | | | |
| Mg | 0.25 | .093 | | | |
| Mn | 0.21 | 0.21 | | | |
| Mo | 310 | 320 | 244 | | 310 |
| N | <200 | <84 | <4 | | 85 |
| Na | 0.016 | 0.0083 | | | |
| Nb | <59 | <63 | <80 | | 60 |
| Nd | <.0077 | 0.0092 | | | |
| Ni | 9.5 | 9.7 | <12 | | |
| O | <610 | <320 | <190 | | 310 |
| Os | <.0088 | <.0072 | | | |
| P | 37 | 29 | <61 | | <30 |
| Pb | <.0075 | <.0061 | | | |
| Pd | <.13 | 0.14 | | | |
| Pt | <.28 | <.19 | | | |
| Rb | <.20 | <.22 | | | |
| Re | <.0037 | <.0044 | | | |
| Rh | <2.3 | <2.9 | | | |
| Ru | <1.4 | <1.3 | <7 | | |

Table 1. Chemical Analyses of 500 kg Heat (832665) of V-4C4-4Ti (wt. ppm)

| Element | GDMS Evans | GDMS Evans | GDMS ORNL | ICP-emission ORNL | Ingot Wah Chang |
|---------|---------------|---------------|--------------|----------------------|--------------------|
| S | 18 | 15 | 40 | | <10 |
| Sb | 0.17 | 0.17 | | | |
| Sc | <.012 | 0.011 | | | |
| Se | <1.7 | <.28 | | | |
| Si | 890 | 680 | %0.1 | <0.3 | 780 |
| Sn | 0.26 | 0.22 | | | |
| Sr | <56 | <61 | <60 | | |
| Ta | <18 | <20 | <3. int | | |
| Te | <0.86 | <.079 | | | |
| Ti | %4.1 | %4.0 | %5.3 | %4.1 | %3.92 |
| Th | 0.013 | 0.0029 | | | |
| U | 0.085 | 0.082 | | | |
| V | bal | bal | bal | | |
| W | 25 | 25 | 22.8 | | |
| Y | <3.8 | <4.5 | <7 | | |
| Zn | 0.11 | 0.12 | | | |
| Zr | <42 | <50 | <65 | | |

Table 2. Chemical Analyses of T Series 15 kg Heats (wt. ppm)

| Element | V-3Cr-3Ti (Heat T91) | | V-4Cr-4Ti-Si (Heat T89) | | V-6Cr-3Ti (Heat T92) | | V-6Cr-6Ti (Heat T90) | | V-5Cr-5Ti (Heat T87) | |
|---------|-------------------------|-------|----------------------------|-------|-------------------------|-------|-------------------------|-------|-------------------------|-------|
| | Ind.** | TWCA* | Ind.** | TWCA* | Ind.** | TWCA* | Ind.** | TWCA | Ind.** | TWCA* |
| B | 3.8 | | 3.5 | | 3.4 | | 3.9 | | | <5 |
| C | <140 | 120 | <120 | 112 | <92 | 105 | <120 | 104 | | 109 |
| N | <5.5 | 62 | <1.8 | 79 | <1.8 | 95 | <4.5 | 85 | | 90 |
| O | <270 | 230 | <270 | 270 | <200 | 280 | <260 | 250 | | 380 |
| Al | 240 | 200 | 270 | 200 | 300 | 255 | 270 | 235 | | 160 |
| Si | 1200 | 940 | 1100 | 1050 | 1000 | 950 | 1400 | 960 | | 545 |
| P | 26 | <50 | 24 | <50 | 22 | <50 | 24 | <50 | | <30 |
| S | 27 | 12 | 23 | 10 | 21 | 12 | 20 | 10 | | <20 |
| Ti | 3.1% | 3.02% | 5.3% | 4.14% | 3.0% | 2.94% | 4.6% | 5.98% | | 5.06% |
| V | 94% | bal. | 90% | bal. | 91% | bal. | 91% | bal. | | bal. |
| Cr | 3.0% | 2.85% | 4.9% | 3.7% | 6.2% | 5.91% | 4.3% | 5.74% | | 4.94% |
| Mn | 1.1 | | 1.9 | | 1.7 | | 1.7 | | | |
| Fe | 110 | 130 | 140 | 170 | 140 | 165 | 120 | 195 | | 160 |
| Ni | 7.6 | | 12 | | 8.3 | | 10 | | | |
| Cu | 79 | 83 | 50 | 83 | 130 | 140 | 48 | 55 | | 67 |
| Zn | 5.3 | | 4.7 | | 4.5 | | 4.0 | | | |
| Ga | 3.9 | | 5.9 | | 7.6 | | 5.0 | | | |
| As | 1.7 | | 2.4 | | 1.4 | | 2.0 | | | |
| Sr | <76 | | <120 | | <70 | | <110 | | | |
| Y | <5.9 | | <9.3 | | <6.1 | | <8.6 | | | |
| Zr | <48 | | <63 | | <56 | | <66 | | | |
| Nb | £55 | <50 | £56 | <50 | £50 | <50 | £54 | <50 | | <100 |
| Mo | 380 | | 360 | | 380 | | 340 | | | 515 |
| Rh | 0.024 | | 0.016 | | 0.02 | | 0.0083 | | | |
| Ru | 0.22 | | 0.18 | | 0.25 | | 0.252 | | | |
| Hf | 0.45 | | 0.12 | | 0.45 | | 0.82 | | | |
| Ta | <9.7 | | <5.2 | | <9.0 | | <98 | | | <36 |
| W | 32 | | 32 | | 32 | | 18 | | | <5 |

*Teledyne Wah Chang analysis

**Analysis obtained by ORNL from an independent vendor

Table 3. Loading List and Identification Codes for Specimens Prepared at ORNL

| Alloy | Specimen Type | Final Heat Treatment | Identification Codes | No. of Specimens |
|---------------------------------|---------------|----------------------|----------------------|------------------|
| V-4Cr-4Ti 832665 | DCT | 1000°C/2 h | WC01-WC10 | 10 |
| | DCT | 950°C/2 h | WC16-WC18 | 3 |
| | PCVN | 1000°C/2 h | WB32, WB35-43 | 10 |
| | PCVN | 950°C/2 h | WB61-WB63 | 3 |
| | MCVN | 1000°C/2 h | WB72-WB77 | 6 |
| | SS-3 | 1000°C/2 h | WE01-WE12 | 12 |
| | TEM | 1000°C/1 h | WG15-WG24 | 10 |
| | TEM | 950°C/1 h | WG31-WG40 | 10 |
| | SS-3 | 1050°C/1 h | WE21-WE25 | 5 |
| | TEM | 1050°C/1 h | WG26-WG30 | 5 |
| GTA Weld V-4Cr-4Ti 832665 | DCT | 950°C/2 h | WE01-WE04 | 4 |
| | MCVN | as machined | WD36-WD38 | 3 |
| | MCVN | 950°C/2 h | WD26-WD31 | 6 |
| | SS-3 | as machined | WF08-WF11 | 4 |
| | SS-3 | 950°C/2 h | WF01-WF04 | 4 |
| EB Weld V-4Cr-4Ti 832665 | DCT | 950°C/2 h | WE11-WE14 | 4 |
| | MCVN | 950°C/2 h | WD51-WD56 | 6 |
| | SS-3 | 950°C/2 h | WF16-WF19 | 4 |
| V-4Cr-4Ti-Si T-89 | DCT | 1050°C/2 h | YA01 | 1 |
| | SS-3 | 1050°C/2 h | YC01-YC03 | 3 |
| | TEM | 1050°C/1 h | YD01-YD10 | 10 |
| V-3Cr-3Ti T-91 | DCT | 1050°C/2 h | XA01 | 1 |
| | SS-3 | 1050°C/2 h | XC01-XC03 | 3 |
| | TEM | 1050°C/1 h | XD01-XD10 | 10 |
| V-6Cr-3Ti T-92 | DCT | 1050°C/2 h | ZA01 | 1 |
| | SS-3 | 1050°C/2 h | ZC01-ZC03 | 3 |
| | TEM | 1050°C/1 h | ZD01-ZD10 | 10 |
| V-6Cr-6Ti T-90 | DCT | 1050°C/2 h | UA01 | 1 |
| | SS-3 | 1050°C/2 h | UC01-UC03 | 3 |
| | TEM | 1050°C/1 h | UD01-UD10 | 10 |

Table 4. Loading List and Identification Codes for Specimens Prepared at ANL

| Alloy | Specimen Type | Final Heat Treatment | Identification Codes | No. of Specimens |
|-----------------------------------|---------------|----------------------|-----------------------|------------------|
| V-4Cr-4Ti 832665 | MCVN 30 | 1000°C/2 h | BL71W: 28, 29, 31, 35 | 4 |
| | MCVN 30 | 950°C/2 h | BL71W: 44, 47, 48, 53 | 4 |
| | PCVN 30 | 1000°C/2 h | BL71W: 19, 24, 26, 32 | 4 |
| | SS-3 | 1000°C/1 h | 71 | 6 |
| | SS-3 | 1000°C/2 h | 71 2H | 6 |
| | SS-3 | 950°C/2 h | 71: A-L | 12 |
| | TEM | 1000°C/2 h | 71 | 10 |
| V-4Cr-4Ti 832665 Laser Weld | MCVN 30 | 400°C/1 h | BL71: A01-A05 | 5 |
| | DCT-A | 400°C/1 h | BL71: A12, A14, A15 | 3 |
| | SS-3 | 400°C/1 h | 71LZ | 4 |
| V-5Cr-5Ti T87 | MCVN 30 | 1000°C/1 h | BL72: A, B, C, D | 4 |
| | SS-3 | 1000°C/1 h | 72 | 8 |
| | TEM | 1000°C/1 h | 72 | 10 |
| V-4Cr-4Ti BL47 | MCVN 30 | 1000°C/1 h | 47: 06, 07, 08 | 3 |
| | PCVN 45 | 1000°C/1 h | 47: 1-5 | 5 |
| | DCT-B | 1000°C/1 h | 47: 07, 08 | 2 |
| | TEM | 1000°C/1 h | 47 | 10 |
| V-4Cr-4Ti-B BL70 | MCVN 30 | 1000°C/1 h | 70: A, B, C, D | 4 |
| | DCT-A | 1000°C/1 h | 70 | 2 |
| | SS-3 | 1000°C/1 h | 70 | 8 |
| | TEM | 1000°C/1 h | 70 | 10 |
| V-4Cr-4Ti-RF BL69 | SS-3 | 1000°C/1 h | 69 | 2 |
| | TEM | 1000°C/1 h | 69 | 10 |

Table 5. Fabrication History for V-4Cr-4Ti Heat 832665: Plate Material

Cast ingot
 ↓
 S.S. jacket/extrude at 1150°C
 to 63.5 mm thick bar
 ↓
 Vacuum anneal, 1050 to 1070°C, 2 h
 Cross roll at 400°C in multiple steps to plate thicknesses of
 12.7, 6.35, 3.81, and 1.02 mm:
 vacuum annealing at 1050 to 1070°C for 2 hr after each 50% reduction
 ↓
 Cold roll 3.81 mm plate to 1.02 mm plate

Plate Identification for ORNL Specimens, V-4Cr-4Ti (832665)

| ORNL I.D. | Manufacturers I.D. | Thickness (mm) | Condition |
|-----------|--------------------|----------------|-----------------------|
| L150 | Plate B | 3.81 | 1050°C, 2 h (TWCA) |
| M150 | Plate A | 3.81 | 1050°C, 2 h (TWCA) |
| P250 | Unannealed | 6.35 | 50% warm-rolled |
| N40 | — | 1.02 | 50% cold-rolled |

Table 6. Fabrication History for Compositional Variants T89, T90, T91, T92

Cast ingot (11.7 mm dia)
 ↓
 S.S. jacket/extruded at 1175 to 1190°C
 ↓
 Vacuum annealed 1075°C, 2 h
 ↓
 Cross-rolled at 400°C in multiple steps to 6.35, 3.81 mm thick
 with vacuum anneals, 1050 to 1075°C, 2 h
 after each 50% reduction
 ↓
 Cross-rolled at RT to 1.02 and 0.76 mm thick
 with vacuum anneals at ~1060°C, 2 h
 after each 50% reduction

Table 7. Fabrication Summary for ORNL Specimens from V-4Cr-4Ti (832665)

| Specimen Type | Fabrication Procedure | Identification Code |
|---------------|---|---------------------|
| MCVN PCVN | Plate M150 (TWCA, annealed, 1050°C) Machined L-T orientation Vacuum annealed 1000°C, 2 h Pre-cracked (UCSB) | WB series |
| DCT | Plate M150 (TWCA, annealed) Machined T-L orientation Vacuum annealed, 1000° or 950°C, 2 h Pre-cracked, side-grooved | WC series |
| SS-3 | Plate N40 (50% C.R., 1.02 mm) Vacuum annealed, 950°C, 2 h Warm-rolled to 0.76 mm (25%) Vacuum annealed, 950°C 2 h Machined Vacuum annealed, 1000 or 1050°C, 2 h | WE series |
| TEM | Plate N40 (50% C.R., 1.02 mm) Vacuum annealed, 1000°C, 1 h Cold-rolled to 0.46 mm (55%) Vacuum annealed, 1000°C, 1 h Cold-rolled to 0.26 mm Punch disks Vacuum annealed, 1050, 1000, or 950°C for 1 h | WG series |

Table 8. Fabrication Summary for V-4Cr-4Ti GTA Weld Specimens

| Specimen Type | Fabrication Procedure | Identification Code |
|---------------|---|---------------------|
| DCT | 50% warm-rolled plate P250; 6.35 mm Full penetration weld No. GTA 9 Machined notch for crack propagation in fusion zone parallel to weld axis Vacuum annealed 950°C, 2 h Pre-cracked; side-grooved Vacuum annealed, 400°C, 1 h | WE series |
| MCVN | 50% warm-rolled plate P250; 6.35 mm Full penetration weld No. GTA 8 Machined notch for crack propagation in fusion zone parallel to weld axis Vacuum annealed, 950°C, 2 h | WD series |
| SS-3 | 50% warm-rolled plate P250; 6.35 mm Full penetration weld No. GTA 8 Machined perpendicular to weld axis with fusion zone in center of gage | WF series |

Table 9. Fabrication Summary for V-4Cr-4Ti EB Weld Specimens

| Specimen Type | Fabrication Procedure | Identification Code |
|---------------|---|---------------------|
| DCT | 50% warm-rolled plate P250; 6.35 mm Full penetration weld No. EBW 9 Machined for crack propagation in weld zone parallel to weld axis Vacuum annealed, 950°C, 2 h Pre-cracked; side-grooved | WE series |
| MCVN | 50% warm-rolled plate P250; 6.35 mm Full penetration weld No. EBW 9 Machined for crack propagation in weld zone parallel to weld axis Vacuum annealed, 950°C, 2 h | WD series |
| SS-3 | 50% warm-rolled plate P250; 6.35 mm Full penetration weld No. EBW 9 Machined perpendicular to weld axis with fusion zone in center of gage Vacuum annealed, 950°C, 2 h | WF series |

Table 10. Fabrication History for ORNL Specimens from Heats T89, 90, 91, 92

| Specimen Type | Fabrication Procedure | Identification Code |
|---------------|---|-----------------------|
| DCT | 50% warm-rolled plate P250; 6.35 mm Machined T-L orientation Vacuum annealed, 1050°C, 2 h Fatigue pre-cracked; side-grooved | UA, YA, XA, ZA series |
| SS-3 | 50% cold-rolled sheet; 0.76 mm Machined L direction Vacuum annealed, 1050, 2h | UC, YC, XC, ZC series |
| TEM | 50% cold-rolled sheet; 0.76 mm Vacuum annealed, 1000°C, 1 h Cold-rolled to 0.25 mm Punched disks Vacuum annealed, 1050°C, 1 h | UD, YD, XD, ZD series |