

GA-A22551

**PRODUCTION AND FABRICATION
OF VANADIUM ALLOYS FOR THE RADIATIVE
DIVERTOR PROGRAM OF DIII-D
— Semiannual Report Input for 1997**

**by
W.R. JOHNSON, J.P. SMITH, and P.W. TRESTER**

DATE PUBLISHED: APRIL 1997

GA-A22551

**PRODUCTION AND FABRICATION
OF VANADIUM ALLOYS FOR THE RADIATIVE
DIVERTOR PROGRAM OF DIII-D
— Semiannual Report Input for 1997**

by
W.R. JOHNSON, J.P. SMITH, and P.W. TRESTER

**Work supported by
Contract No. DE-AC03-89ER51114
for the San Francisco Operations Office
U.S. Department of Energy**

**GENERAL ATOMICS PROJECT 3896
DATE PUBLISHED: APRIL 1997**

**PRODUCTION AND FABRICATION OF VANADIUM ALLOYS FOR THE
RADIATIVE DIVERTOR PROGRAM OF DIII-D
— SEMIANNUAL REPORT INPUT FOR 1997**

W.R. Johnson, J.P. Smith, and P.W. Trester (General Atomics)

SUMMARY

V-4Cr-4Ti alloy has been selected for use in the manufacture of a portion of the DIII-D Radiative Divertor upgrade. The production of a 1200-kg ingot of V-4Cr-4Ti alloy, and processing into final sheet and rod product forms suitable for components of the DIII-D Radiative Divertor structure, has been completed at Wah Chang (formerly Teledyne Wah Chang) of Albany, Oregon (WCA). Joining of V-4Cr-4Ti alloy has been identified as the most critical fabrication issue for its use in the RD Program, and research into several joining methods for fabrication of the RD components, including resistance seam, friction, and electron beam welding, is continuing. Preliminary trials have been successful in the joining of V-alloy to itself by electron beam, resistance, and friction welding processes, and to Inconel 625 by friction welding. An effort to investigate the explosive bonding of V-4Cr-4Ti alloy to Inconel 625 has also been initiated, and results have been encouraging. In addition, preliminary tests have been completed to evaluate the susceptibility of V-4Cr-4Ti alloy to stress corrosion cracking in DIII-D cooling water, and the effects of exposure to DIII-D bakeout conditions on the tensile and fracture behavior of V-4Cr-4Ti alloy.

PROGRESS AND STATUS

1. Introduction

General Atomics (GA) has developed a plan for the utilization of vanadium alloys in the DIII-D tokamak. The plan is being implemented with the assistance of the Argonne, Oak Ridge, and Pacific Northwest National Laboratories (ANL, ORNL and PNNL), and will culminate in the operation of a water-cooled vanadium alloy structure in the DIII-D Radiative Divertor (RD) upgrade.^{1,2} The use of a vanadium alloy will provide a meaningful step towards developing advanced materials for fusion power applications by 1) demonstrating the in-service behavior of a vanadium alloy (V-4Cr-4Ti) in a typical tokamak environment, and 2) developing knowledge and experience on the design, processing, and fabrication of full-scale vanadium alloy components.

The program currently consists of three phases: first, small vanadium alloy specimens and coupons are being exposed inside the DIII-D vacuum vessel to evaluate the effects of the DIII-D vacuum environment; second, a small vanadium alloy component will be designed, manufactured, and operated in conjunction with the existing DIII-D divertor; and third, the upper private flux baffle of the new double-null, slotted divertor will be designed, fabricated from vanadium alloy product forms, and installed in DIII-D. A major portion of the program is research and development to support fabrication and resolve key issues related to environmental effects. The plan is being carried out by GA as part of the DIII-D Program, and with the support of ANL, ORNL, and PNNL, participants in the Materials Program of the Department of Energy's Office of Fusion Energy (DOE/OFE).

2. PHASE 1: Specimen and Coupon Exposures and Analysis

Miniature Charpy V-notch (CVN) impact and tensile specimens of V-4Cr-4Ti alloy (ANL 500 Kg heat)³ were exposed/monitored in DIII-D in a position on the vessel wall behind the divertor baffle for ~9 months.⁴ The maximum temperature experienced by the specimens during vacuum bakeouts was ~350°C. GA has collated the environmental data, and ANL has developed and is now implementing an analysis plan

for the evaluation of the specimens.⁵ CVN tests conducted at temperatures over the range of -196 to $+150^{\circ}\text{C}$ indicated ductile behavior for all test temperatures. Tensile tests conducted at ambient and elevated temperatures indicated values similar to unexposed material. A new set of V-4Cr-4Ti alloy samples, installed during the January 1996 vent, is currently undergoing exposure to provide more data for confirmation and statistical verification.

In parallel to these exposures, additional V-4Cr-4Ti alloy samples underwent short-term exposures utilizing the DIII-D Divertor Material Exposure System (DiMES). A V-4Cr-4Ti alloy disc, and subsequently CVN specimens, were exposed/monitored during bakeout of the DIII-D vacuum vessel.⁴ The maximum temperature experienced by the specimens during vacuum bakeouts was also $\sim 350^{\circ}\text{C}$. GA has collated the environmental data, and ANL has developed and is now implementing an analysis plan to determine the pickup of any impurities in these specimens.⁵ CVN tests conducted at temperatures over the range of -196 to $+150^{\circ}\text{C}$ have indicated ductile behavior for all temperatures. A surface analysis of the V-4Cr-4Ti alloy disc specimen has been completed at Sandia National Laboratory in Albuquerque, NM, and evaluation of that data is being performed at ANL. Other DiMES exposures of V-4Cr-4Ti alloy are being considered to evaluate the effects of other DIII-D environmental conditions (e.g., discharge cleaning, boronization, etc.).

3. PHASE 2: Small Component Exposure

A second step in the DIII-D Vanadium Plan is to install a small V-4Cr-4Ti alloy component in DIII-D. This component will be manufactured utilizing many of the methods proposed for the Phase 3 water-cooled private flux baffle. The component will be installed, exposed for some period of tokamak operations, and then removed. Samples from the component will be excised, and metallurgical analyses and property measurements will be made on the excised materials. Specific plans have not yet been defined.

4. PHASE 3: Radiative Divertor Program

The design, manufacture, and installation of a V-4Cr-4Ti alloy private flux baffle structure for the upper divertor of the RDP will be performed as the third phase of the vanadium alloy program. Details of the structural design of the toroidally-continuous, water-cooled and radiatively-cooled structural panels which make up the V-4Cr-4Ti alloy PFB were presented in a previous report.⁶

The water-cooled panels for the V-4Cr-4Ti alloy baffle will be fabricated of sandwich construction from two 4.8 mm sheets, each containing a wide 1.5 mm deep slot milled into its face to provide an internal coolant channel. Resistance seam welding is the primary candidate process being considered for structurally joining the panel edges, with electron beam welding proposed for making a leak-tight perimeter seal. Other methods of joining are proposed for different areas of the design such as the graphite armor tile attachment studs and cooling water inlet and outlet manifolds. To facilitate installation, V-4Cr-4Ti alloy/Inconel 625 joints are planned to provide a bi-metallic tube nipple. This component will be used to transition V-alloy water-cooled panels to Inconel 625 tubes which provide a cooling water supply.

A. Fabrication of Product Forms For The Radiative Divertor

Processing of the V-4Cr-4Ti alloy by WCA was initiated in September 1995. Prior to this reporting period, the alloy had been processed into a 1200 kg ingot, extruded into two (2) billets (A and B1), and machined/ground into sheet bar material. Details of these stages of the processing were presented in previous reports.^{4,6}

During July 1996, full chemical analyses of the sheet bar materials, A and B1 (samples from both ends), were completed. The analyses for both materials were within specification limits except for Nb (87–92 ppm

measured; <20 ppm specified), Ag (<1–3 ppm measured; <1 ppm specified), and Ca, Na, K, and Mg (<5 ppm, 1–3 ppm, 3–4 ppm, and <10 ppm, respectively, measured; <1 ppm specified for each), and were accepted. The two pieces of sheet bar (10.4 cm × 23.4 cm × 245.1 cm and 107.4 cm, respectively) were heated to 400°C and longitudinally rolled into 4.7 cm thick × 23.4 cm wide plate (~540 cm and ~240 cm long, respectively). Plate from sheet bar A (Plate A) was cut into seven (7) 60.3 cm long sections, one (1) 67.3 cm long section, and one (1) 39.7 cm long section. Plate from sheet bar B1 (Plate B1) was cut into three (3) 60.3 cm long sections and one (1) 44.4 cm long section.

The ten (10) 60.3 cm long sections from both plates (designated A1, A2, A3, A5, A6, A7, A8, B1A, B1C, and B1D via their relative locations in the two plates) were cross-rolled after heating to 400°C, and successively reduced to 4.8 mm thick × ~65 cm wide by ~185 cm long sheets. Several 400°C rolling cycles, with intermediate vacuum anneals of 2 hours at ~1050°C, were used. The sheets were then trimmed to ~59 cm wide × ~178 cm long, roller leveled after heating to 315°C, cleaned and pickled, and final vacuum annealed for 2 hours at 1000°C. The final annealing treatment was selected based on an annealing study performed at GA on edge trim material taken from one of the as-rolled sheets (sheet B1C). The GA study indicated that recrystallization of the material was complete after 1 to 2 hours at 1000°C, with a final grain size of ~ASTM 6–8. A metallographic analysis (at WCA) of samples taken from both ends of a full-length piece of edge trim material (from sheet A2), which was production annealed along with the ten sheets, indicated 98% recrystallization of the material, with a final average grain size of ASTM 8. Chemical analyses (for H, O, N, and C only) were performed on samples taken from each of the finished sheets. The values obtained were within specification limits except for O values for two sheets, B1C and B1D (400–410 ppm measured; <400 ppm specified), and were accepted. The values were close to the in-specification values obtained previously for extruded billet material, and were also within the accuracy of the analysis method relative to the specification limit for O.

The 67.3 m long section from Plate A (A4), taken from the middle of the sheet bar, was cut into several 4.7 cm × 4.7 cm × 67.3 cm long sections, and machined to a diameter of ~4 cm. This material was swaged after heating to 400°C and successively reduced to 11 mm diameter rod in several 400°C swaging cycles. Intermediate vacuum anneals of 2 hours at ~1050°C were used between swaging cycles. The rods were then cut into fifteen (15) 180 cm lengths, straightened, centerless ground to 10.1 mm diameter, cleaned and pickled, and final vacuum annealed for 2 hours at 1000°C. The final annealing treatment was again selected based on an annealing study performed at GA on a sample of as-swaged rod. The GA study indicated that complete recrystallization of the material, with a final grain size of ~ASTM 5–7, occurred at 1000°C after 1 to 2 hours. Metallographic analysis (at WCA) of samples taken from the ends of one representative production-annealed rod length indicated 95%–98% recrystallization of the material, with a final average grain size of ASTM 6. Chemical analyses (for H, O, N, and C only) were performed on samples taken from three (3) representative finished rod lengths. The values obtained were within specification limits except for O values for one rod sample (420 ppm measured; <400 ppm specified), and were accepted. Like those out-of-specification values for sheet material, this value was also close to the in-specification values obtained previously for extruded billet material, and was also within the accuracy of the analysis method relative to the specification limit for O.

The finished sheet and rod product forms were received by GA from WCA in October 1996. Support will be provided by ANL and ORNL in the development of basic engineering design properties for these product forms.

B. Manufacturing Technology Development

As manufacturing development is a major focus of this project a significant amount of research and development is being performed in this area. The RD structure will require many metal/metal joints, and

joining development is therefore a key area of study. GA is using private IR&D funds to complement welding efforts at ANL and ORNL, investigating several different joining processes which are attractive for fabrication of RD components including resistance seam, friction, electron beam welding, and most recently, explosive bonding. The scope of the GA joining development efforts has been limited by the availability of material, some of which has been purchased from WCA (V-5Cr-5Ti; Heat ID #932394) and some of which has been supplied by ANL (V-5Cr-5Ti alloy; Heat ID #832394 and V-4Cr-4Ti alloy; Heat ID #832665³).

Resistance Welding Studies

Resistance seam welding studies in air are continuing to develop the method for structurally joining sheets of vanadium alloy together to make the water-cooled panels for the RDP.

As reported previously,⁶ spot weld trials were performed on 4.8 mm thick V-5Cr-5Ti alloy sheet material producing diffusion bonds at the lowest power levels utilized and weld nuggets at higher power levels. Representative weld trial samples demonstrated considerable strength (~685 MPa based on weld nugget diameter only, and ~380 Mpa based on total bonded diameter, i.e., diffusion bond + weld nugget) in simple spot weld lap shear tests even though they typically contained single, small, centrally-located pores. The lower strength values reported (380 MPa) were still greater than that specified by American Welding Society standards for material of this strength level. Additional spot weld trials were performed on this material using increased electrode (forge) pressures at the end of the weld cycle in an attempt to minimize the previously-observed porosity. Although the porosity was not completely eliminated, it was substantially reduced.

Additional confirmatory spot weld trials have recently been performed on 4.8 mm thick V-4Cr-4Ti alloy sheet procured by GA for the DIII-D Radiative Divertor program. Using power settings similar to those utilized for the previous trials on V-5Cr-5Ti alloy, successful joints were also produced, again ranging from diffusion bonds to fully-melted weld nuggets. Single spot lap shear tests on welded joints demonstrated strengths which were similar to those developed previously for V-5Cr-5Ti alloy (~380 Mpa). In addition, room-temperature strengths obtained for diffusion-bonded samples, i.e., samples with no detectable melting at the faying surfaces, were observed to be substantially the same as for those which developed fully-melted nuggets, thus demonstrating that successful high strength spot weld joints in V-alloy could be obtained in air, and over a wide range of power levels (from diffusion bonds to welds).

Resistance seam (overlapping spots) welds were then performed on similar 4.8 mm V-4Cr-4Ti alloy sheet using copper alloy wheel electrodes and a variety of power settings, and electrode (wheel) pressures, wheel diameters, and wheel radii. Although full melting (i.e., nugget formation) was not achieved at the faying surfaces of the coupon samples, seam diffusion bonds (~5 to 10 mm in width) were obtained which exhibited room-temperature lap shear test strengths comparable to that achieved for similar spot-welded (and spot-bonded) material. A vacuum leak test of a sectioned ~75 mm (~30 overlapping spot welds) long seam-bonded sample indicated a helium leak rate of $<1 \times 10^{-11}$ std. cc/s, well below that required for DIII-D components. Attempts at producing fully-melted nuggets, i.e., welds, at the faying surfaces, by utilizing very high power inputs, resulted in some melting of the copper electrodes, infiltration of copper into the alloy, and surface cracking.

In addition to the resistance welding studies, preliminary tests have been completed to study the potential for stress corrosion cracking of vanadium alloy resistance welds in DIII-D cooling water. A potential for slow crack growth under static and dynamic loading conditions was identified for the crevice created between RD water-cooled panel plates via the structural resistance seam weld to be utilized during

component fabrication. PNNL has performed several crack growth tests in DIII-D cooling water on specimens of V-4Cr-4Ti alloy (ANL 500 kg heat³) to investigate this potential issue.⁷

Typical operating conditions expected for the V-alloy RD component, including stress and temperature cycles, were provided to PNNL in order to select the type of test and test conditions to be imposed. Two room-temperature crack growth tests were performed on compact tension specimens exposed under a static load condition to DIII-D water. GA computer analyses/calculations indicated a very small stress intensity factor ($\sim 3 \text{ ksi}\sqrt{\text{in.}}$) could be operable in the vicinity of a seam weld crevice, resulting from possible crevice-opening loads in this area (water pressure in the panel). Since very little extension of this crevice (flaw) was expected at this value based on the known behavior of similar materials, crack growth tests were performed at a stress intensity of $30 \text{ Ksi}\sqrt{\text{in.}}$, a factor of ten times the calculated value. Two tests were conducted, one for 32 days and another for 60 days. No measurable crack growth was observed during either test. This result was confirmed by SEM analysis of fracture surfaces obtained after the specimens were rapidly broken in tension.

Friction Welding Studies

Two types of friction welding trials are in progress. Inertia (shop) and portable friction welding have been selected as candidate processes for joining vanadium alloy to itself, and inertia welding has been selected for creating a bi-metallic joint between vanadium alloy and Inconel 625. Initial rod-to-rod inertia weld trials of vanadium alloy (V-5Cr-5Ti alloy) bonded to itself, and to Inconel 625, were successful in air, without any protective environment. Metallography showed complete bonding with no indications of porosity or cracking, and mechanical tests at room temperature resulted in failures in material well away from the joint and HAZs.

As reported previously,⁶ inertia weld trials were performed to fabricate bi-metallic V-5Cr-5Ti alloy/Inconel 625 joints in a butt joint configuration which could be machined into a bi-metallic tube nipple, a component which could be joined to a V-alloy water-cooled panel (via a controlled shop weld, e.g., electron beam welding) and subsequently to an Inconel 625 cooling water tube (via a field tungsten-inert gas weld). These trials, although encouraging, were not completely successful, producing joints which were not vacuum leak tight during helium leak checking and with variable strengths in room-temperature tensile tests (~ 70 to 380 MPa). Metallography of a typical sample revealed that bonding could be substantially enhanced by a modified joint configuration/design.

Additional trials were performed using the new joint configuration, and were successful. Metallography of a representative sample indicated complete bonding with no porosity. A number of representative trial samples were then machined for checking the vacuum leak tightness of the joints and their strengths. All samples were observed to be vacuum leak tight to DIII-D standards (down to $4.4 \times 10^{-8} \text{ Torr}$) and exhibited strengths $> \sim 720 \text{ MPa}$ in room-temperature tensile pull tests, with failures occurring in a ductile (tearing) mode in the V-alloy away from the joint. This strength level is considerably higher than the ultimate tensile strength of the V-alloy ($\sim 520 \text{ MPa}$), and is believed to result from a combination of increases in the strength of the V-alloy near the joint due to the heavily hot-worked structure (fine grain size) developed in this vicinity by the welding process, and the presence of the notch from the transition of the smaller diameter joint area to the larger diameter threaded region for the specimen grips.

As previously reported,⁶ preliminary portable friction welding trials of vanadium alloy rod to plate were performed to develop methods of *in-situ* replacement of studs on water-cooled panels. Initial trials achieved substantial bonding, but the hardness of the weld interface increased significantly, signaling the development of a brittle joint. In addition, a large amount of grain growth occurred at the interface and substantial heat-affected zones (HAZs) were developed. It was also noted during the trials that the

temperature of the weld interface was significantly higher than that observed in the inertia weld process, and the time to create the weld was considerably longer.

Additional friction welding trials were performed using parameters [higher ram (rod) pressures and ram rotational speeds] which were expected to minimize the excessive heat input observed in previous samples. As a starting point, welding (ram) pressures were selected to be similar to those previously used in successful rod-to-plate inertia (in-shop friction) welds of V-alloy,⁴ and welding times were reduced to decrease the heat input to the joint and minimize the development of HAZs near the joint interface. Metallographic evaluation of selected samples from these trials indicated porosity-free bonding over ~100% of the contacting areas and thinner HAZs compared to previous trial samples. The HAZ thickness generally decreased with increased ram pressure for a given ram speed. A single room-temperature tensile pull test performed on a sample processed using an intermediate ram speed failed at or near the joint at a stress value of ~345 MPa, approximately equivalent to the tensile yield strength of the base V-alloy.

Electron Beam Welding Studies

Preliminary electron beam welding trials on 6.35 mm V-5Cr-5Ti alloy sheet have been completed, and were reported previously.⁶

Explosive Bonding Studies

Explosive bonding have been investigated as an alternate method for producing a dissimilar metal joint between V-alloy and Inconel 625 for application as a tube nipple/connector. This connector would be used to transition a V-alloy water-cooled RD panel to an Inconel 625 water supply tube. A lap joint for this tube-to-tube configuration may be more preferable for this application compared to the butt joint configuration developed by inertia welding because of its enhanced mechanical strength and potential for a longer (up to ~10 times) leak path between the cooling water inside the tube and the external vacuum environment of DIII-D.

A preliminary explosive bonding trial of a V-4Cr-4Ti alloy tube to an Inconel 625 round bar was performed. Dye penetrant inspection of specimens sectioned along the length of the ~150 mm long (22.1 mm o.d. × 2 mm wall tube) processed trial sample indicated good bonding and no interface porosity at the end opposite the explosive charge detonation end of the sample, and minimal bonding at the detonation end. Metallography of the sample confirmed that bonding (with no porosity) had been achieved over about 1/2 of the length of the sample, the interface between the V-4Cr-4Ti alloy and Inconel 625 along the length of the sample exhibiting the serrated structure typical of explosively-bonded metals. The joint was also leak tight, a helium leak check of a ~12 mm length section of the sample indicating a helium leak rate of $\sim 6 \times 10^{-10}$ std. cc/s, well below required DIII-D standards. Strength tests of the joint and additional bonding trials are planned. Complete bonding over the entire length of a trial sample is expected to be achievable by the addition of more explosive material on the outer surface of the V-4Cr-4Ti alloy tube.

CONCLUSIONS

A program for utilizing vanadium alloys in DIII-D has been developed to progress the deployment of low activation alloys for fusion applications, and production of material for this program has been completed. A 1200 kg V-4Cr-4Ti alloy ingot of acceptable chemical composition has been melted, extruded into rectangular billets, and processed into sheet and rod product forms. Preliminary successes have been achieved in developing similar and dissimilar metal welds in vanadium alloy by resistance, inertia, portable friction, and electron beam welding, and explosive bonding methods.

ACKNOWLEDGEMENTS

This is a report of work supported by the U.S. Department of Energy under Contract No. DE-AC03-89ER51114, and General Atomics under internal R&D funding.

REFERENCES

- ¹J. P. Smith, W. R. Johnson, R. D. Stambaugh, P. W. Trester, D. Smith, and E. Bloom, "Vanadium Alloys For The Radiative Divertor Program of DIII-D," to be published in *Proc. of the 16th IEEE/NPSS Symposium on Fusion Engineering*, Champlain, Illinois, September 30 through October 5, 1995.
- ²J. P. Smith, W. R. Johnson, R. D. Stambaugh, P. W. Trester, D. Smith, and E. Bloom, "Utilization of Vanadium Alloys In The DIII-D Radiative Divertor Program," to be published in *Proc. of the 7th International Conf. on Fusion Reactor Materials (ICFRM-7)*, Obninsk, Russia, September 25–29, 1995.
- ³H. M. Chung, H.-C. Tsai, D. L. Smith, R. Peterson, C. Curtis, C. Wojcik, and R. Kinney, "Fabrication of 500-kg Heat of V-4Cr-4Ti," in *Fusion Reactor Materials Semiannual Progress Report*, DOE/ER-0313/17, Oak Ridge National Laboratory, Oak Ridge, Tennessee (1994), p. 178–187.
- ⁴W. R. Johnson, J. P. Smith, and R. D. Stambaugh, "Production and Fabrication of Vanadium Alloys for the Radiative Divertor Program of DIII-D", in *Fusion Reactor Materials Semiannual Progress Report*, DOE/ER-0313/19, Oak Ridge National Laboratory, Oak Ridge, Tennessee (1995), p. 5–11.
- ⁵H. Tsai, D. L. Smith, W. R. Johnson, and J. P. Smith, "Performance of V-4Cr-4Ti Material Exposed to the DIII-D Tokamak Environment", to be published in *Fusion Reactor Materials Semiannual Progress Report*, DOE/ER-0313/21, Oak Ridge National Laboratory, Oak Ridge Tennessee (1997).
- ⁶R. J. Kurtz and W. R., "An Evaluation of the Susceptibility of V-4Cr-4Ti to Stress Corrosion Cracking in DIII-D Cooling Water", to be published in *Fusion Reactor Materials Semiannual Progress Report*, DOE/ER-0313/21, Oak Ridge National Laboratory, Oak Ridge Tennessee (1997).
- ⁷W. R. Johnson, J. P. Smith, and R. D. Stambaugh, "Production and Fabrication of Vanadium Alloys for the Radiative Divertor Program of DIII-D", in *Fusion Reactor Materials Semiannual Progress Report*, DOE/ER-0313/20, Oak Ridge National Laboratory, Oak Ridge, Tennessee (1996), p. 3–10.