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PRODUCTION AND FABRICATION OF VANADIUM ALLOYS FOR THE RADIATIVE DIVERTOR PROGRAM OF DIII–D
— Annual Report Input for 1995

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W.R. JOHNSON, J.P. SMITH, and R.D. STAMBAUGH

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

SUMMARY

V-4Cr-4Ti alloy has been recently selected for use in the manufacture of a portion of the DIII–D Radiative Divertor modification, as part of an overall DIII–D vanadium alloy deployment effort developed by General Atomics (GA) in conjunction with the Argonne and Oak Ridge National Laboratories (ANL and ORNL). The goal of this work is to produce a production-scale heat of the alloy and fabricate it into product forms for the manufacture of a portion of the Radiative Divertor (RD) for the DIII–D tokamak, to develop the fabrication technology for manufacture of the vanadium alloy Radiative Divertor components, and to determine the effects of typical tokamak environments on the behavior of the vanadium alloy. The production of a ~1300-kg heat of V-4Cr-4Ti alloy is currently in progress at Teledyne Wah Chang of Albany, Oregon (TWCA) to provide sufficient material for applicable product forms. Two unalloyed vanadium ingots for the alloy have already been produced by electron beam melting of raw processed vanadium. Chemical compositions of one ingot and a portion of the second were acceptable, and Charpy V-Notch (CVN) impact tests performed on processed ingot samples indicated ductile behavior. Material from these ingots are currently being blended with chromium and titanium additions, and will be vacuum-arc remelted into a V-4Cr-4Ti alloy ingot and converted into product forms suitable for components of the DIII–D RD structure. Several joining methods selected for specific applications in fabrication of the RD components are being investigated, and preliminary trials have been successful in the joining of V-alloy to itself by both resistance and inertial welding processes and to Inconel 625 by inertial welding.

PROGRESS AND STATUS

1. Introduction

General Atomics (GA), along with the Argonne and Oak Ridge National Laboratories (ANL and ORNL), has developed a plan for the utilization of vanadium alloys in the DIII–D tokamak which will culminate in the fabrication, installation, and operation of a vanadium alloy structure in the DIII–D Radiative Divertor (RD) modification.[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 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 consists of three phases: first, small vanadium alloy coupons is being exposed in DIII–D at positions in the vessel floor and behind the existing divertor structure; second, a small vanadium alloy component will be operated in conjunction with the existing divertor, and third, during the forthcoming RD modification, in 1998 a portion of the upper section of the new double-null, slotted divertor will be fabricated from vanadium alloy product forms. A major portion of the program is research and development to support fabrication and resolve key issues related to environmental effects. The execution of the plan is a joint effort by GA, the DIII–D Program, and Department of Energy (DOE) Material Program participants, primarily ANL and ORNL.

On the basis of excellent properties that have been determined for both laboratory-scale and production-scale heats, V-4Cr-4Ti alloy has been identified as the most promising vanadium-based candidate alloy for application in fusion reactor structural components,[3,4] and has been selected for procurement in product forms applicable for the manufacture of a portion of the DIII–D RD modification.

The alloy has been, and is currently being exposed in DIII–D during various stages of DIII–D operation to assess the effects of a typical tokamak environment on the behavior of the alloy. Procurement of product forms (sheet and rod) of the alloy has also been initiated, and a ~1300-kg V-4Cr-4Ti alloy ingot is currently in processing at TWCA to provide applicable product forms for the manufacture of a portion of the upper section of the DIII–D radiative divertor. In addition, fabrication studies are in progress to develop joining methods applicable to manufacture of the vanadium alloy RD components.

2. PHASE 1: Specimen and Coupon Exposures and Analysis

In March of 1995, minature Charpy V-notch (CVN) impact and tensile specimens of V-4Cr-4Ti alloy from a large production-scale heat fabricated for ANL by TWCA (500-kg, Heat ID#832665)[4] were installed in DIII–D in positions behind the divertor baffle. These specimens were scheduled for long-term exposure (~1 year). ANL supplied the samples, and GA provided the design and fabrication of the hardware for retaining the samples. The specimen environment was monitored (i.e., thermocouples, residual gas analyzer, and pressure gauges) during various stages of DIII–D operation (e.g., baking, discharge cleaning, boronization, plasma discharges, etc.). Comparison of specimen and vessel thermocouple readings indicate that the specimens closely followed (within ~5°C) the outer vessel wall temperature (i.e., the inner wall of the outer toroidal surface of the vessel). These samples were removed during the January 1996 DIII–D vent, having experienced numerous thermal cycles up to temperatures of ~350°C as well as exposure to trace amounts (10^{-9}–10^{-6} atm) of potentially embrittling impurities (e.g., H_2, O_2, H_2O, N_2, B, CO, CO_2, and a number of hydrocarbons). GA is currently analyzing the environmental data while ANL is performing an evaluation of the specimens. These data will be used to provide a preliminary assessment of the effects of a tokamak environment on the behavior of the alloy. During the January 1996 vent, additional V-4Cr-4Ti alloy CVN and tensile specimens were installed in the DIII–D specimen holder, and will be exposed (and monitored) until the next DIII–D vent.

In parallel, other V-4Cr-4Ti alloy (Heat ID #832665) samples were exposed in a position in the DIII–D vessel floor utilizing the DIII–D Divertor Material Exposure System (DiMES). Utilization of the DiMES allows for an exposure of a material without waiting for a vent to retrieve samples. A V-4Cr-4Ti alloy disc was exposed during the initial baking and cleaning of DIII–D after the February 1995 vent, as a post-vent baking cycle representing what is expected to be the most potentially severe environmental condition for the vanadium alloy specimen. During the exposure the disc was monitored using an infrared camera (temperature), RGA, and pressure gauges. It was removed soon after the bake/clean was completed having experienced temperatures and exposure to trace amounts of impurities typical of bakeout conditions. The disc is currently being evaluated at ANL to quantify any possible impurity(s) pickup. Additional exposures of five (5) miniature V-4Cr-4Ti alloy CVN specimens to a similar baking cycle were performed in July 1995, and are also currently under evaluation at ANL. Other DiMES exposures are planned 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 which will operate in conjunction with the existing DIII–D Advanced Divertor. This component will be a single radiatively-cooled divertor plate or a small representative water-cooled component which will be operated in series with the primary water-cooled divertor panels. The component will be installed during an upcoming DIII–D vent, exposed for some period of operation, and then removed. Samples from the component will be excised by GA, and GA, ANL, and ORNL will perform metallurgical analyses and property measurements on the excised materials. A decision as to the specific component has not been made at this time.

4. PHASE 3: Radiative Divertor Program

The final step in the deployment of a vanadium alloy in DIII–D is the design, manufacture, and installation of a portion of the upper half of the RD structure using V-4Cr-4Ti alloy, with the other portions being fabricated from Inconel 625. The V-4Cr-4Ti alloy structure will contain toroidally-continuous, water-cooled structural panels with inertially-cooled graphite tiles mechanically attached to their surfaces by welded studs. The panels will be water cooled during machine operation, experiencing a maximum temperature of ~60°C. During post-vent clean-up, hot air will replace the water in the coolant channels of the structure, and the structure (along with the DIII–D vessel) will be baked to ~400°C. The panels will be supported from the vacuum vessel by Inconel 718 supports which will provide the required strength for reacting disruption loads and the flexibility for withstanding differential thermal growth during baking. Due to the lower electrical resistivity of the V-4Cr-4Ti alloy as compared to Inconel 625, the toroidal current flow during disruption will be approximately 4 times larger. The design of the panels and supports will be modified to react the larger loads.[1]

The panels will be made in segments and fabricated of sandwich construction from two 4.76 mm sheets, each containing a 1.5 mm deep coolant channel milled into its side. Resistance seam welding is the primary candidate process being considered for joining the panel edges and creating a leak tight seal. Electron beam welding may be used in addition to resistance seam welding as a process for ensuring the leak tightness of the water-cooled panels. Electron beam welding is being considered for making the water connections (V-4Cr-4Ti alloy tubing) to the panels with inertial welding as a possible backup. Inertial welding is the primary candidate process being considered for joining studs to the panels for graphite tile attachment, and for making V-4Cr-4Ti alloy to Inconel 625 bi-metallic joints for effecting in-vessel gas tungsten arc (GTA) field welds to Inconel 625 water supply tubes.

4.1 Fabrication of Product Forms For The Radiative Divertor

The production campaign for the V-4Cr-4Ti alloy product forms consists of electron beam melting of unalloyed vanadium ingot materials, alloying with high purity Cr and Ti and vacuum-arc melting (two times) of a large-scale (~1300-kg) V-4Cr-4Ti alloy ingot, extrusion of the alloyed vanadium ingot into rectangular bars, and subsequent conversion by rolling (and drawing) and heat treatments into 4.76 mm thick sheet and 10.16 mm diameter rod product forms. The specification for the alloy was developed by GA with input from ANL and ORNL. Particular attention was given to the control of impurities to meet the immediate goals for the RD program and also future goals for further deployment of vanadium alloys in advanced fusion systems including the minimization of Nb, Mo, and Ag for low neutron activation; the optimization of Si (400–1000 ppm) to suppress neutron-induced swelling; and the limiting of O, N, C, Cu, S, P, Cl, Ca, K, Mg, Na, and B to avoid grain boundary segregation and precipitation of embrittling phases.[4] The final specification of unalloyed vanadium and alloyed vanadium ingot chemistries for the V-4Cr-4Ti alloy procurement is listed in Table 1.

Processing of the V-4Cr-4Ti alloy by TWCA was initiated in September 1994. The raw vanadium for the unalloyed vanadium ingots for the alloy, in the form of ~100-kg lots was prepared from high purity vanadium oxide by the aluminothermic process. Chemical analysis of the ~30 lots processed, although deemed not specifically precise with respect to predicting the composition of subsequent ingots, revealed substantial variation in their chemical compositions. Several key chemical attributes were noted as follows: 1) all of the lots were out of specification limits with respect to Si (400–1000 wppm), containing only 100–200 wppm, a variation often observed in processed raw vanadium due to the varying Si levels typically found in the starting material (vanadium oxide); 2) all of the lots contained <50 wppm Mo (within specification limits); 3) approximately half of the lots contained <50 wppm Nb, with the other half containing substantially more Nb (several hundred wppm); and 4) lot-averaged values for Fe and S were slightly greater than specifications limits. Variations in other elements were also noted with most elements being within specification limits for all of the lots. Rather than process additional raw vanadium in the hopes of increasing the Si to within specification levels (400–1000 wppm), and delay the production of the V-4Cr-4Ti alloy, or make Si additions to the vanadium during processing of the alloyed vanadium ingot, and run the risk of producing unwanted Si segregation in the alloy, the low-Si raw vanadium lots were accepted for further processing.

Lots of raw vanadium were then selected into two groups by TWCA in collaboration with GA based on their overall chemistries with respect to meeting all of the specification requirements, and two 395 mm diameter vanadium ingots were processed by electron beam melting. Chemical analysis of the resulting ingots indicated that both ingots were generally within specification limits for all elements except primarily for Nb (See Table 1). One ingot (~900-kg) had a Nb level of which averaged ~40 wppm and another larger ingot (~2200-kg) had a Nb level of several hundred wppm. Samples (~0.5 in. x ~0.5 in. x ~1.5 in.) were excised from the surfaces of the low-Nb ingot and the higher-Nb ingot portion at their mid-lengths for mechanical property measurements to confirm their purity (and microstructure). These samples were subsequently processed by cold rolling and annealing at ORNL, and were then machined into CVN specimens and tested at ANL at –196°C and above to evaluate their toughness (ductility), a property which is extremely sensitive to impurities in vanadium. Both ingot materials exhibited ductile behavior and had impact toughness (ductility) values similar to that obtained by ANL for the pure vanadium ingot material for the 500-kg V-4Cr-4Ti alloy heat.[5] Since the blending of the low-Nb ingot with a portion of the higher-Nb ingot was expected to result in only a doubling of the final Nb level, from ~40 wppm to ~90 wppm Nb, and this increase was not expected to compromise the properties of the alloy for its intended application, a decision was made to blend the ingot materials to produce the ~1300-kg of the alloy.

TABLE 1
CHEMICAL COMPOSITION GOALS FOR V-4Cr-4Ti ALLOY AND CHEMISTRIES OF VANADIUM INGOTS

Content, parts/million by weight (ppm)*				
Element	**Specification For Unalloyed Vanadium Ingot**	**Unalloyed Vanadium Ingot [Heat ID #820645] (Average of 3 Measurements)**	**Unalloyed Vanadium Ingot [Heat ID #820642] (Average of 3 Measurements)**	**Specification For Alloyed Vanadium (V-4Cr-4Ti) Ingot**
Cr	–	-	-	4±0.5 wt %
Ti	–	-	-	4±0.5 wt %
Si	400–1000	173	197	400–1000
H	<10	<3	<3	<10
O	<400	313	213	<400
N	<200	113	153	<200
C	<200	24	25	<200
Al	<200	243	167	<200
Fe	<300	147	135	<300
Cu	<50	<50	<50	<50
Mo	<50[†]	<50	<50	<50[†]
Nb	<20[†]	40	226	<20[†]
Cl	<3	‡	‡	<3
Ga	<10	<5	<5	<10
Ca	<1[†]	<10[d]	<25[d]	<1[†]
Na	<1[†]	<5[d]	<5[d]	<1[†]
K	<1[†]	<5[d]	<5[d]	<1[†]
Mg	<1[†]	<10[d]	<10[d]	<1[†]
P	<30	<30	<30	<30
S	<30	<10	<10	<30
B	<5	<5	<5	<5
Ag	<1	<5[d]	<5[d]	<1
V	balance	balance	balance	balance

[*]Unless otherwise specified in weight %.

[†]Desired values — <5 ppm Mo, <1 ppm Nb, <1 ppm total Ca+Na+K+Mg.

[‡]Not analyzed.

[#]Request initiated to TWCA for re-analysis to higher sensitivity level

During February 1996, consolidation of the two vanadium ingot materials with each other was initiated at TWCA, to be followed by addition of high purity Cr and Ti (double vacuum-melted Ti), and alloying by vacuum arc melting (twice) of the V-4Cr-4Ti alloy ingot.

4.2 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. In addition to the welding development work on vanadium alloys being conducted at ANL and ORNL (laser, electron beam, GTA, etc.) to support this program, GA is investigating several different joining processes which are attractive for fabrication of RD components including resistance seam, electrodischarge (stud), inertial, and electron beam welding. The scope of the GA joining development efforts has been limited by the availability of material, some of which has been purchased from TWCA (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 #832665). Support is also being provided by ANL and ORNL in the development of basic engineering design properties.

Resistance Welding Studies

Resistance seam welding is planned to form the closure weld in the RD water-cooled structural panels. Industrial companies with seam welding experience have been contacted and resistance spot welding trials are currently being performed by one of these vendors (K-T Aerofab of El Cajon, CA).

Resistance welding trials (in air) were initially performed on sandwiches of 1 mm pure and alloyed vanadium sheet by B-J Enterprizes of Albany, Oregon. Weld nuggets were formed and microhardness measurements on a sectioned sample showed very little increase (<10%) in hardness in the weld or heat-affected zones (HAZ) over the parent metal.

Thicker sheet material (3.81 mm) of V-4Cr-4Ti was obtained from ANL and resistance spot weld trials (12 trials) were initiated at the local vendor. Although some diffusion bonding (up to ~80% of the interface regions directly under the weld electrodes) was obtained between sheet sandwich samples, no weld nuggets were observed. A second set of trials (4 trials) using different weld parameters (i.e., slightly higher current inputs) were made over several of the original spots from the first trials. Diffusion bonding was observed to a greater extent (~95%), yielding strengths of greater than 135 MPa using crude shear strength tests, but still no weld nuggets were developed. A third set of trials (8 trials) was performed with similar V-4Cr-4Ti alloy sheet material using weld parameter data obtained from the literature for carbon steel which were found to have similar strength and resistivity to V-4Cr-4Ti. These trials were successful, with metallography of sectioned samples indicating good diffusion bonds for the lowest current trials, good weld nuggets with no porosity for intermediate current levels, and weld nuggets with some porosity (single central pores) for the highest current levels. Weld nuggets for the best trials were ~7 mm in diameter, and microhardness measurements showed less than ~10% increase in hardness in the weld and HAZs over that of the parent metal. Additional spot weld trials are now in planning using material similar in thickness to that to be used for the RD water-cooled panels (4.76 mm). V-5Cr-5Ti alloy sheet material, 6.35 mm in thickness, was supplied by ANL, and has recently been cold rolled by ORNL to this thickness for these trials.

Inertial Welding Studies

Inertial welding to join a vanadium alloy (V-5Cr-5Ti alloy) to itself, and to stainless steel and Inconel 625, have been investigated. A vendor with experience in joining pure vanadium to Monel was located (Interface Welding of Carson, CA) and preliminary welding trials have been completed. Inertial welding trials on V-5Cr-5Ti alloy to itself (19.05 mm diameter disc to 9.52 mm diameter rod) were successful. Metallography showed complete bonding with no indications of porosity or cracking. Microhardness measurements showed only slight increases in hardness in the weld and HAZs. Tensile pull tests were performed on three weld trial specimens at room temperature, and all three samples failed in the threaded grip area of the V-5Cr-5Ti alloy rod (at ~520 MPa stress) well away from the weld areas. Torque tests were also performed on two additional samples to measure the capability of the joint to withstand torsion loads as would be experienced in a stud-to-plate joint. Again, failures occurred in the V-5Cr-5Ti alloy threaded sections away from the weld joints. Trials to join stainless steel (19.05 mm diameter disc) to V-5Cr-5Ti alloy (9.52 mm diameter rod) did not achieve complete bonding, but results indicated that development of such bi-metallic joints could be enhanced by decreasing the diameter of the stainless steel part and enhancing its forgability relative to that of the softer vanadium alloy. Joining trials were continued on Inconel 625 (12.7 mm diameter rod) to V-5Cr-5Ti alloy (9.52 mm diameter rod) in order to be more relevant to the planned DIII–D effort. Additional trials using similar size V-5Cr-5Ti alloy rod (9.52 mm diameter) and smaller diameter (6.35 mm diameter) Inconel 625 rod to match the forgabilities of the two materials were successful. Metallography showed complete bonding with no porosity or cracking. Tensile pull tests were performed on three weld trial samples. Two samples failed in the Inconel 625 section well away from the weld area (at ~930 MPa stress) and one sample failed at the approximate weld interface, but at a stress level of ~760 MPa. Additional inertial weld trials are now being planned to fabricate bi-metallic tube joints.

Stud Welding Studies

Stud welding trials utilizing the drawn arc method were performed between vanadium alloy (V-5Cr-5Ti) stud (9.6 mm diameter) and plate (3.8 mm thickness) materials. A representative from the stud welding equipment manufacturer (TRW Nelson Stud Welding Division of Walnut, CA) assisted in the trials. A range of welding parameters were used without success. The weld materials would not stay in the weld area, blowing out from the sides. Cracking was also observed (audible) during cool down of the materials. Visible bonding never exceeded ~50%, and for many of the trials achieved much less than this value. A second set of trials was performed with a different stud geometry, but were also unsuccessful. These failures, and the successes achieved in the vanadium alloy inertial weld trials, prompted the investigation of portable friction welding as a process for the attachment of studs. Preliminary V-5Cr-5Ti alloy stud-to-plate, portable friction welding trials were performed at RamStud Inc. of Smyrna, GA, and substantial bonding was achieved. Evaluation of these trials is currently in progress.

Electron Beam Welding Studies

Preliminary electron beam welding trials have also been initiated at GA. Initial weld parameters were obtained from ORNL personnel and weld penetration tests were performed using 6.35 mm thick V-5Cr-5Ti alloy plate acquired from ANL. Initial metallography was performed on these weld penetration trial samples to establish specific weld parameters for creating a lap weld of two 3.85 mm thick vanadium alloy sheet materials (V-5Cr-5Ti alloy to V-4Cr-4Ti alloy). A lap weld of the materials was created and metallurgically examined. Good weld penetration was obtained with no indications of cracking. Microhardness measurements showed less than ~10% increase in hardness. Tensile and shear specimens are planned to evaluate the strength of the welds.

CONCLUSIONS

A program for utilizing vanadium alloys in DIII-D has been developed, and production of material for this program has started. Two vanadium ingots have been electron beam melted as base materials for a ~1300-kg V-4Cr-4Ti alloy ingot. Chemical analyses of one ingot (900-kg) and a portion (~400-kg) of another larger (~2200-kg) ingot, Charpy V-notch impact test results on material excised from both ingots were found to be satisfactory to continue the processing of the V-4Cr-4Ti alloy. Preliminary successes were achieved in developing similar metal weldments of vanadium alloys by resistance spot and inertial welding methods, and in producing vanadium alloy/Inconel 625 dissimilar metal weldments by inertial welding.

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