



Gas tungsten arc welding of vanadium alloys with impurity control

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Abstract

Gas tungsten arc welding in vanadium alloys is controlled by interstitial impurities. Techniques have been developed to weld V–4Cr–4Ti in a high-purity argon atmosphere resulting in a DBTT of -20 °C. The atmosphere was controlled by a Zr–Al getter which is activated at high temperature to obtain a clean surface then cooled and allowed to absorb hydrogen and oxygen impurities. Through the use of low-oxygen base metal and high-purity weld filler wire, a DBTT of -145 °C was obtained. Experiments using electron beam welding have shown that grain size also has an important effect on weld ductility. Introduction of nitrogen and yttrium has been used to study their effect on grain size. Using a combination of atmosphere control, alloy purity control, and grain size control, it is anticipated that V–Cr–Ti alloys will be weldable in field conditions.

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1. Introduction

Although vanadium alloys offer advantages for fusion reactors such as low neutron activation and high thermal conductivity, a fusion device cannot be constructed unless the alloys can be successfully welded. Welding can be accomplished using techniques that are more commonly identified with laboratory scale welding such as electron beam welding and high-purity glove box arc welding [1]. However, despite the success of these techniques, welding must be accomplished in factories and in field construction environments. It is the goal of this investigation to develop techniques for gas tungsten arc (GTA) welding and adapt them for use in actual fabrication conditions. This study focuses on three areas for refinement: atmosphere control, metal impurity control, and grain size control in the V–4Cr–4Ti alloy.

Perhaps the most important factor in analyzing interstitial behavior is to differentiate between impurities,

particularly oxygen, in solid solution and impurities existing as precipitates. This difference has been demonstrated by precipitation post-weld heat treatments [2]. Using a high vacuum heat treatment of 950 °C/2 h, the interstitial concentration was unchanged, but oxygen-rich precipitates, determined to be $\text{Ti}_{16}(\text{O}_3\text{N}_3\text{C}_2)$, formed which reduced the concentration of oxygen in the lattice [2]. Prior to achieving ultra-pure atmospheres, and thus low-interstitial welds, reductions in DBTT (ductile–brittle transition temperature) in excess of 200 °C were achieved through the above precipitation heat treatment [2]. As higher purity atmospheres were used, and welds with lower DBTT were made, the precipitation heat treatment produced progressively smaller decreases in the DBTT. A post-weld heat treatment is not to be neglected as a means of improving weld properties since the highest purity atmosphere achievable in the laboratory cannot necessarily be achieved in the field. Although arc welding is expected to be the primary method for welding the large structures involved in a fusion reactor, some components, especially those fabricated in a factory, might employ laser welding. Laser welding has an added advantage that it can accomplish a post-weld heat treatment by scanning the weld with a

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defocused beam, making a furnace heat treatment unnecessary [3].

A second source of interstitial impurities in solution is the impurities present in the base metal and weld wire prior to welding. During welding some of the precipitates dissolve and introduce the interstitials into solution where they again harden and embrittle the lattice. For this reason, higher purity base metal and high-purity weld wire were investigated [4]. This paper reports some of the results of these two thrusts for improving vanadium alloy weld behavior.

2. Experimental methods

Gas tungsten arc welding in a controlled atmosphere glove box was the general approach used for the test welds. Welds were typically made on plates 6.4 mm in thickness since thick plates of vanadium alloys are more difficult to weld than thin sheets, and this is about the largest thickness expected for to be used in first wall and blanket structures. Plates were machined to form a 75° included angle V-groove with a 2.4 mm root opening. Manual multi-pass welds of 6–11 passes were made using filler wire made from alloys to be tested. Further details of the welding chamber are found in Ref. [1].

To conduct an investigation on pre-existing impurities, similar plates of V–4Cr–4Ti were produced in Japan with an oxygen level of 180 wt ppm (NIFS Heat-1). In addition, high-purity welding wire was produced with an oxygen level of only 36 wt ppm [4,5]. Test welds were made using the same techniques and apparatus. For controls, welds were made using the US materials without opening the glove box between welds.

Atmosphere control was accomplished by evacuation of the box to the range of 10^{-4} Pa and back filling with high-purity argon (99.999%). However, this atmosphere, without gettering, was found to be unsatisfactory for vanadium welding.¹ Various getter systems were tested with a cold getter being the most effective. The getter consisted of a Zr–Al alloy (SAES no. St 101) applied to a constantan substrate that served to conduct a heating current.¹ The getter was heated to 700 °C to allow impurities to diffuse into the metal to expose a fresh surface. The getter proved effective for hydrogen and oxygen. Atmospheric control was monitored by a residual gas analyzer (RGA).² A small turbomolecular pumped chamber housing the RGA head was separated from the welding chamber by a bleed valve so that gas could be continuously sampled while maintaining a high vacuum for operation of the RGA. A disadvantage of this system was slow response because of travel time

from the welding chamber to the RGA. Nonetheless, changes in the composition of the atmosphere could be monitored as the weld progressed.

Charpy impact testing was employed as a sensitive test of the quality of the weld (DBTT measurement). It is a rapid test that served the purpose of the experiment. A miniature Charpy specimen, commonly used for irradiation testing, was chosen. The specimen was 25.4 mm in length and 3.33 mm on a side with a notch making a 30° included angle machined to a depth of 0.66 mm with a root radius of 0.05–0.10 mm. The specimens were machined such that the crack propagated longitudinally along the weld, entirely within the fusion zone. The heat-affected zone will be tested in a later phase of the program, once the desired properties of the fusion zone are obtained.

3. Results

3.1. Atmospheric control

The first step in interstitial control is to control the welding atmosphere. Early tests using conventional shielding gas were totally unacceptable. A high-purity argon glove box was used in a series of experiments where the atmosphere was made progressively more pure with the addition of getter systems to remove oxygen and, finally, hydrogen as well. Table 1 shows the results of three welds made with progressively higher purity atmospheres. The total oxygen in the weld changes little, but the oxygen in solution is expected to be significantly different. A significant improvement in DBTT is shown with increasing atmospheric purity; however, the hydrogen concentration increased as the oxygen level decreased. To separate the effects of oxygen and hydrogen, Charpy specimens were outgassed at 400 °C/1 h to remove hydrogen. The dramatic reduction in DBTT is shown in Table 1, indicating the importance of both oxygen and hydrogen.

Removal of hydrogen from specimens GTA 16 and 17 not only reduced the DBTT but also introduced a high-temperature minimum in the Charpy energy curves. This behavior is attributed to a loss of ductility that is restored by the onset of twinning as the test temperature is reduced [1]. High purity, low temperature, large grain size, and high strain rate all favor twinning in vanadium alloys [6,7]. With introduction of the cold getter system, high-purity welds could be obtained with the DBTT below –20 °C. A typical curve appears in Fig. 1 where the high-temperature minimum is apparent.

3.2. Alloy impurity control

A series of tests was conducted on alloys prepared with initially low concentrations of interstitial

¹ Manufactured by SAES Getters, Milan, Italy.

² Model 100 C, manufactured by UTI, Sunnyvale, CA, USA.

Table 1
Impurity levels and DBTT for welds in V–4Cr–4Ti

Weld	PWHT	Atmosphere (wt ppm)		Weld fusion zone (wt ppm)			DBTT
		Oxygen	Moisture	Oxygen	Nitrogen	Hydrogen	
GTA 13	None	4	23	374	104		57
GTA 13	950 °C/2 h						60
GTA 16	None	0.8	25	370	107	63	85
GTA 16	400 °C/1 h						38
GTA 17	None	≪1	<1	347	99	99	20
GTA 17	400 °C/1 h					1.9	0

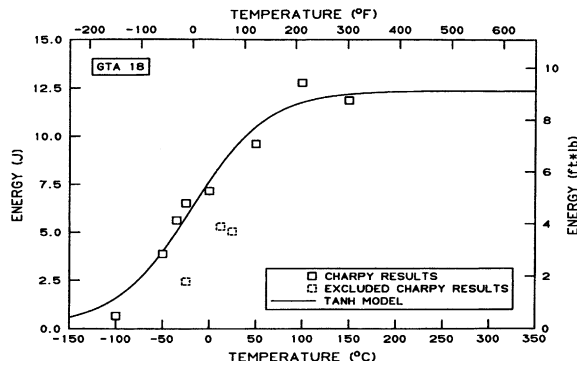


Fig. 1. Charpy energy curve for a GTA weld in V–4Cr–4Ti using a getter system. The dashed symbols are believed to result from twinning.

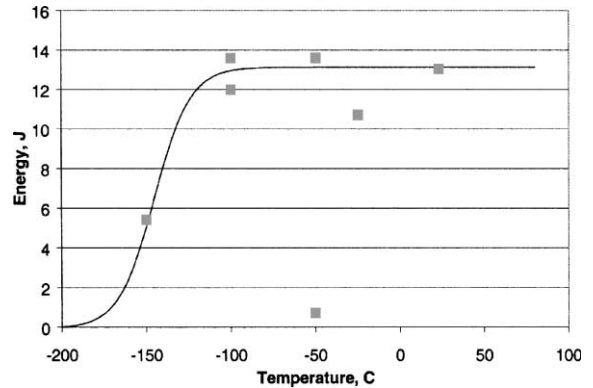


Fig. 2. Charpy energy curve for a GTA weld in low-oxygen V–4Cr–4Ti using high-purity weld filler metal.

impurities [4]. The results, shown in Table 2, clearly demonstrate that not all impurities in solid solution arise from the atmosphere. Using the Japanese V–4Cr–4Ti base metal, JPN, with the high-purity Japanese V–4Cr–4Ti filler metal, JPNHP, a DBTT of -145 °C was achieved, although the high-temperature minimum is still cause for concern, Fig. 2. This weld will have greater tolerance to radiation hardening and the consequent shift in DBTT. The effect of base metal is illustrated by VGTA 23, VGTA 24, VJGTA 3, and

VJGTA 5 which use US base metal of heats 832665 and 832864, similar heats of V–4Cr–4Ti. Using similar welding atmospheres, all have higher DBTT levels than those using the low-oxygen base metal (Table 2). The welds VJGTA 3 and VJGTA 5 demonstrate a very significant observation that high-purity filler metal alone can result in a weld with a low DBTT, although not as low as if both base metal and filler metal were of high purity. By removing hydrogen through a $400\text{ °C}/1\text{ h}$ outgassing heat treatment, the DBTT was reduced by

Table 2
Weld series investigating base metal and weld wire initial purity effects (concentrations in wt ppm)

Weld	Base–filler	Base metal			Filler metal			Atmosphere		Weld fusion zone			DBTT (°C)
		O	N	H	O	N	H	O	H ₂ O	O	N	H	
VGTA 23	US–US	310	73	4.3	370	110		0.75	6				6
VGTA 24	US–US	370	120	4.3	370	110		1	2				25
VGTA 24, 400 °C/1 h	US–US	370	120	4.3	370	110		1	2				–20
VJGTA 1	JPN–JPN	180	88		180	110	3.7	0.75	6	198	108	49	–89
VJGTA 2	JPN–JPNHP	180	88		36	89	4.8	0.75	6	73	97	69	–145
VJGTA 3	US–JPNHP	310	73	4.3	36	89	4.8	0.75	6	135		59	–83
VJGTA 5	US–JPN	370	120		180	110		0.7	<2				–40

45 °C. This confirms that this weld was contaminated by hydrogen.

3.3. Grain size control

The effect of grain size on DBTT has been demonstrated by electron beam welding V–5Cr–5Ti. By defocusing the electron beam, a weld with a factor of two larger grain size was achieved, and this resulted in an increase in the DBTT by about 150 °C.

Since nitrogen is known to refine grain size [8], an attempt was made to dope welds with nitrogen to achieve a smaller grain size in the weld fusion zone. Levels of nitrogen were increased from 100 wt ppm to 135 and 350 wt ppm in the fusion zone of GTA welds. There is no discernable reduction in grain size in the weld specimens; however, precipitates appear to decorate subgrain boundaries in the case of the 350 wt ppm weld, Fig. 3. Further experiments will be performed on plates sufficiently thick to fabricate miniature Charpy specimens to determine ductility.

Yttrium was alloyed with V–4Cr–4Ti weld wire to levels of 0.5 and 0.2 wt% in order to investigate the ability of yttrium to refine grain size [9]. Grain size was in fact slightly refined, especially at the higher level. However, there appeared to be an intervening embrittling mechanism associated with the Y so that the DBTT increased to 53 and 13 °C for the welds containing 0.5 and 0.2 wt% Y, respectively, as opposed to –20 °C that was achieved in the absence of Y. Further studies and transmission electron microscopy are required to investigate the source of the embrittlement, but the yttrium is expected to form small precipitates serving to getter oxygen and nitrogen [10].

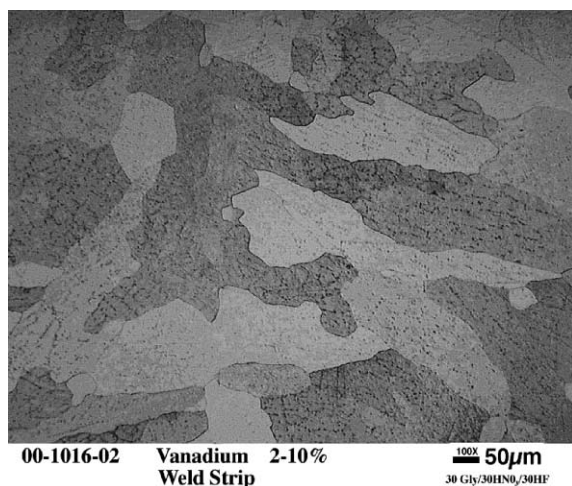


Fig. 3. Gas tungsten arc weld in V–4Cr–4Ti doped with 350 wt ppm nitrogen.

4. Discussion

Interstitial impurities and grain size have been found to be two crucial factors in determining the ductility of welds in V–4Cr–4Ti as measured by Charpy impact testing. This investigation has shown that a major source of contamination by interstitial impurities is the welding atmosphere. Since oxygen diffuses about a factor of 10 faster than nitrogen at 1000 °C, oxygen is usually the more troublesome embrittling agent in vanadium even though nitrogen is at least as potent as a hardening agent [11,12]. The lower diffusivity is evident from vanadium welding experience with atmospheres of varying purity. In such cases oxygen varies between welds made with atmospheres of diverse impurity levels, but nitrogen varies to a much lesser degree [1,2] since it diffuses through the vanadium at a slower rate. Control of oxygen in the welding atmosphere is the first factor that enabled reduction in the DBTT to levels in the range of –20 °C. The next increment of DBTT reduction was achieved by using base metal and filler wires with lower oxygen concentrations. It is hypothesized that in each case, it is oxygen in solid solution that is the damaging species, especially considering the effect of the precipitation heat treatment in reducing the DBTT and the observation that fracture is nearly always entirely transgranular, cleavage below the DBTT. However, if oxygen in solid solution is the dominant hardening and embrittling agent, why can the heat treatment of 950 °C/2 h not reduce the DBTT to –145 °C, the level achieved by the high-purity filler wire in the JPN plate? There are several possibilities. One is that the heat treatment is not sufficiently long to reach equilibrium with the titanium oxycarbonitride precipitates. Another is that the solubility of oxygen in equilibrium with the oxycarbonitride is higher than previously thought. Still another possibility is that the precipitates contribute to the observed embrittlement. DiStefano has studied niobium, in which oxygen has a similar diffusivity at the temperatures of interest and found by internal friction that a heat treatment of 1000 °C/1 h resulted in almost complete precipitation [13]. This supports the hypothesis that the precipitates contribute to hardening, but does not rule out incomplete precipitation or higher solubility in vanadium alloys, in particular in the more complex V–4Cr–4Ti.

Hydrogen was controlled using the Zr–Al getter; nonetheless, the alloy impurity control series was contaminated with hydrogen. Removal of hydrogen from VGTA 24 resulted in a decrease in the DBTT by 45 °C. The experiment illustrates the difficulty in controlling hydrogen but also suggests that an even lower DBTT might be achieved by alloy impurity control and better atmosphere control. The source of the hydrogen and the possibility of contamination following the welding will be further investigated.

5. Conclusions

1. Gas tungsten arc welds in 6.4 mm plate can be made with adequate ductility as shown by the $-145\text{ }^{\circ}\text{C}$ DBTT.
2. Alloy interstitial purity, atmospheric purity, and grain size are all important parameters in vanadium alloy welding.
3. Grain size can be influenced by additions of nitrogen or yttrium, but much research must be done on this area.
4. Relaxation of some welding parameters might result in adequate welds under field conditions.

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