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Development of techniques for welding V–Cr–Ti alloys

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Abstract

Welding vanadium alloys is complicated by interstitial impurity introduction and redistribution at elevated temperatures. Gas tungsten arc (GTA) welding, which will probably be required for the fabrication of large tokamak structures, must be done in a glove box environment. Welds were evaluated by Charpy testing. GTA welds could be made with a ductile to brittle transition temperature (DBTT) of 50°C with a post-weld heat treatment (PWHT) or by using a heated Ti getter system on the glove box to reduce interstitial contamination. Titanium-O,N,C precipitates in the fusion zone were found to transform to a more oxygen-rich phase during a PWHT of 950°C/2 h. Hydrogen was found to promote cleavage cracking following welding in cases where the atmosphere was contaminated. Grain size and microstructure also affected weld embrittlement.

1. Introduction

Since any tokamak structure will have many kilometers of welds, using vanadium alloys will only be possible if methods are found for making sound welds. Moreover, they must be made on large structures under field or modified field conditions. The quality of welds in vanadium alloys is largely determined by the absorption, migration, and precipitation of impurities. Factors such as purity and composition of the welding atmosphere, the base alloy and the filler metal, and time at temperature are all important in determining the properties of the weld.

The present study focuses on the development of gas tungsten arc (GTA) welding of vanadium alloys but uses electron beam welding to support the studies. Electron beam (EB) welding is an ultra clean technique, which allows the effect of impurities from the atmosphere to be separated from other effects such as dissolution of precipitates and grain size effects. However, most welding of the very large components of a tokamak will have to be done by arc welding or perhaps laser welding, both of which will have to be performed without a vacuum chamber, although some form of enclosure will probably be necessary.

2. Experimental methods

Most of the research employed 6.4 mm plate in order to investigate a size typical of first wall structures. Because of the slower cooling rate of a thicker plate, interstitial contamination is expected to be worse than for thin sheet. For supporting tests, 0.76 mm sheet was also used with autogenous GTA welds.

The 6.4 mm plate was machined with a 75° included angle V-groove butt joint positioned with a 2.4 mm root opening. Filler wire made from the same alloy and heat as the base metal was used to make multi-pass welds using direct current, electrode negative at a current range of 100–140 A at 12 V.

Except for initial scoping studies, all GTA welds were made in an argon atmosphere in a glove box which was a 4.7 m^3 stainless steel chamber that could be evacuated to 10^{-4} Pa with a diffusion pump. It was back filled with high purity argon to one atmosphere to enable the use of the gloves. Moisture levels were typically below 40 wt.ppm.

After several welds were evaluated, it was determined that improvements in the glove box atmosphere were required. To reduce the contamination by oxygen, a purification system was added to the glove box. The

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system consisted of a titanium element held at 600–800°C, a molecular sieve trap to reduce moisture, and a circulation pump. The addition of this system permitted oxygen levels below 1 ppm in the atmosphere.

Studies on vanadium alloys have employed a miniature Charpy specimen which has proved useful in determining the fracture properties of this class of alloys [1]. Since fracture and embrittlement appear to be the most important properties to investigate in welds in vanadium alloys, the Charpy specimen was also chosen for the present study. A miniature Charpy impact specimen with a length of 25.4 and 3.33 mm on a side was used. A blunt notch making a 30° included angle was machined to a depth of 0.66 mm.

The study began with the V–5Cr–5Ti alloy (Wah Chang Heat 832394) mostly because of availability of 6.4 mm plate. When V–4Cr–4Ti, a US Fusion Program prime candidate alloy, plate became available, it was used for the welding research. It too was manufactured by Wah Chang of Albany, OR, as Heat 832665 [2]. It was demonstrated that when the V–5Cr–5Ti alloy received a final heat treatment of 950°C for 2 h in processing, the impact properties were similar to those of the V–4Cr–4Ti alloy [1]. For this reason, the information gained from the V–5Cr–5Ti alloy is believed to be valid and applicable.

3. Results and discussion

3.1. Oxygen effects

Welds in the V–5Cr–5Ti alloy demonstrated a very significant effect of post weld heat treatment. As shown in Fig. 1, GTA welding resulted in a ductile to brittle transition temperature (DBTT) above 200°C. After a post-weld heat treatment (PWHT) of 950°C for 2 h in a vacuum with a pressure below 6×10^{-5} Pa, the DBTT was reduced by about 200°C. Although less dramatic, the same effect was observed in the V–4Cr–4Ti alloy. Electron beam welds, which of necessity use a vacuum environment, have resulted in DBTT's of −100°C and even lower without a PWHT [3].

It has been shown in Nb–1Zr that a high-temperature PWHT is necessary to prevent attack by liquid lithium [4]. This phenomenon is understood in terms of oxide precipitation. Oxygen is not only absorbed from the welding atmosphere but is put into solution by re-solution of existing precipitates of zirconium oxide and quenched in solution upon cooling. The PWHT allows precipitation of this oxygen from solution in the form of zirconium oxide. Since the vanadium alloys under study are similar refractory alloys gettered by titanium, similar behavior is anticipated. Indeed the GTA welds of both

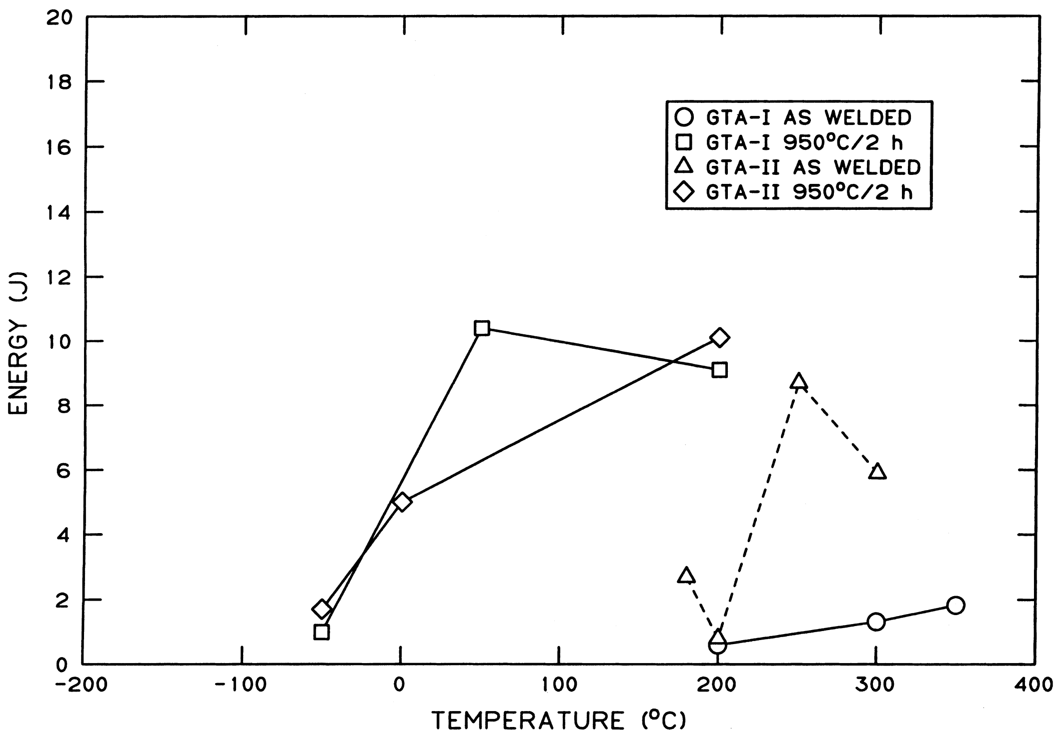

Fig. 1. Charpy impact curves for two GTA welds of V–5Cr–5Ti before and after a post-weld precipitation heat treatment.

vanadium alloys experienced a significant increase in the DBTT following welding, which was improved by a PWHT of 950°C for 2 h.

Transmission electron microscopy identified precipitates as $V_8Ti_8(C_3NO_{.65})$, in the heat affected zone of a GTA weld. After a PWHT of 950°C/2 h, larger precipitates were observed and found to have the composition: $Ti_7(O_4N_2C)$. It is evident that the precipitate is gettering oxygen as a result of the heat treatment and changing composition to a more stable and more oxygen-rich titanium oxycarbonitride. Titanium oxycarbonitride precipitates have been observed previously in V–15Cr–5Ti, V–20Ti, and V–3Ti–1Si by Schober and Braski, although of different stoichiometry, but they reported a large variation in stoichiometry between the vanadium alloys studied [5].

Lower temperature post-weld heat treatments were attempted, but results were inconsistent. In the case of V–5Cr–5Ti, a 750°C PWHT resulted in an increase in the DBTT by about 100°C. In the case of V–4Cr–4Ti, a similar PWHT resulted in a slight decrease in the DBTT. However, the 950°C heat treatment for 2 h gave consistently improved weld properties.

To study the effects of impurities, a series of welds was made with varying levels of impurities. Using the titanium gettering system described previously, an especially pure welding atmosphere was achieved with an oxygen level of 4 wt.ppm and a moisture level of 23 wt.ppm. A high-purity weld, made using the gettering system, and two additional lower-purity welds, made by deliberately raising the impurity levels in the glove box atmosphere, were produced. The welds were designated GTA 13 through GTA 15, and the impurity levels are given in Table 1.

The concentrations of interstitial impurities are also shown in Table 1, and a correlation, although not perfect, is demonstrated between the impurity levels in the atmosphere and the oxygen and nitrogen levels in the metal as determined by inert gas fusion analysis. The influence of impurity levels on the DBTT is also evident with a clear monotonic relationship between atmospheric impurity levels and DBTT. Although embrittlement is sensitive to the level of atmospheric impurities, the internal levels of O and N do not change very much. It is believed that most of the O and N from the atmosphere enters in solution. Since much of the pre-existing O and N is in precipitates, the total concentrations do not change by a large amount, but the levels in solution change by a more significant amount, and it is impurities in solution that cause embrittlement.

Transmission electron microscopy was used to study microstructures in the fusion zones of this series of welds. The highest purity weld, GTA 13, exhibited little precipitation in the as-welded condition in contrast to the least pure, GTA 15, which showed precipitates both in the matrix and on grain boundaries. Following a PWHT of 950°C/2 hr, thin plate-like precipitates were evident in GTA 13 as shown in Fig. 2. Parallel electron energy loss (PEELS) analysis identified the precipitates as Ti_{16} $(O_3N_3C_2)$. This phase was different and more Ti-rich than that discussed previously. More analysis is required on the precipitates; however, the oxygen behavior is consistent in the two cases analyzed in that it precipitates from solid solution during the PWHT. The involvement of oxygen is consistent with the improvement in mechanical properties following the PWHT and in agreement with the previous work on niobium alloys. The least pure weld, GTA 15, also contained contiguous precipitation on grain boundaries, thicker and larger than those observed in the weld prior to the PWHT. The intermediate purity weld, GTA 14, also contained the plate-like precipitates, but sample quality and inhomogeneities in precipitation made it difficult to analyze.

In previous welds fracture morphology was nearly all cleavage for lower shelf Charpy specimens. Fracture surfaces were examined from lower-shelf specimens of the impurity series welds. Consistent with previous welds, the specimens failed nearly entirely by cleavage, and as test temperature increased, some ductile rupture was observed. However, there was a small amount of intergranular fracture, especially in GTA 15, the weld with the precipitate-decorated grain boundaries. Fig. 3

Table 1
Impurity concentrations and DBTT for GTA Welds

Weld	PWHT	Welding atmosphere		Fusion zone concentration			DBTT (°C)
		Oxygen (wt. ppm)	Moisture (wt. ppm)	Oxygen (wt. ppm)	Nitrogen (wt. ppm)	Hydrogen (wt. ppm)	
GTA 13	None	4	23	374	104		57
GTA 13	950°C/2 h						60
GTA 14	None	14	84	352	110	21	82
GTA 14	950°C/2 h						80
GTA 15	None	27	260	412	146	15	228
GTA 15	950°C/2 h						86
GTA 16	None	0.8	25	370	107	63	85
GTA 16	950°C/2 h						38

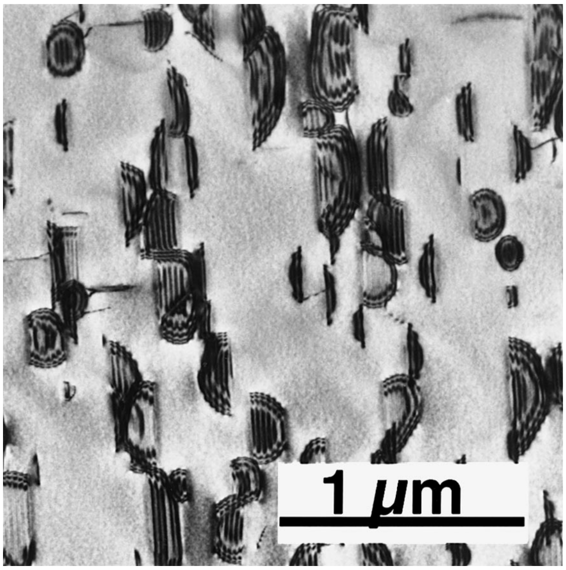

Fig. 2. Plate-like precipitates in Weld GTA 13 in V–4Cr–4Ti after heat treating at 950°C/2 h.

shows a secondary intergranular crack in a Charpy specimen tested at 100°C. In contrast, GTA 13 exhibited very flat cleavage surfaces with secondary cleavage cracks but no intergranular failure. This can be seen in Fig. 4 which shows an intact grain boundary on a cleavage fracture surface in a specimen tested at 50°C. Although the cleavage fracture mechanism predominates in all observed brittle failures, contaminated specimens appear to be weakened by intergranular precipitates. Intergranular separation might produce stress concentrations that could initiate cleavage cracks, and thus contribute to cleavage fracture, but more specimens must be studied to support this conclusion.

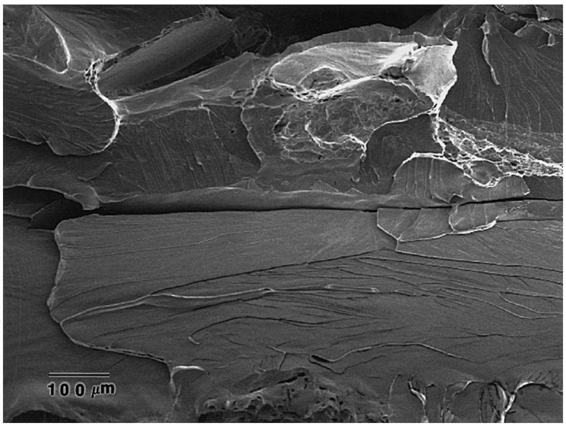

Fig. 3. Fracture surface of Weld GTA 15 in V–4Cr–4Ti showing secondary intergranular cracking.

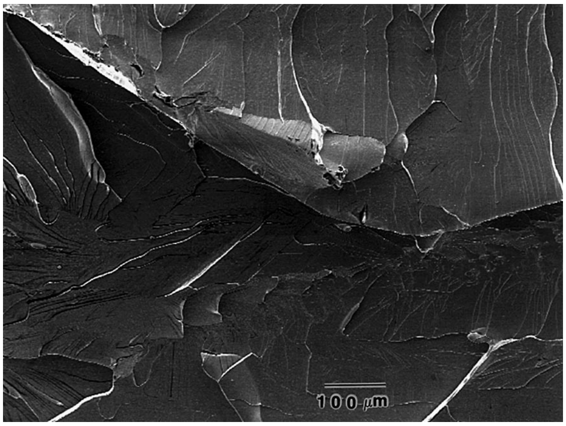

Fig. 4. Fracture surface of Weld GTA 13 in V–4Cr–4Ti showing grain boundary.

Fig. 5 compares Charpy curves for the three welds of the impurity series before and after the PWHT. The effect of the PWHT on the impure weld is rather dramatic with a shift in DBTT of nearly 200°C. The two welds with lower impurity levels, however, show little if any shift in DBTT. The conclusion is that if the atmospheric oxygen level is low initially, the precipitation PWHT is not necessary.

3.2. Hydrogen effects

The results of all of the PWHT studies were complicated by occasional lower shelf fractures at temperatures at which ductility was usually observed. In all cases, the brittle specimens exhibited cleavage fracture. Specimens at temperatures above and below that of the brittle specimen demonstrated ductile behavior. This spurious behavior has been previously observed in welds in vanadium alloys [6]. This observation led to investigation of hydrogen embrittlement in the welds, especially in light of the complete cleavage fracture. Hydrogen was further implicated by a GTA weld that fractured prior to machining specimens and was found to contain 3000 at.ppm hydrogen.

The impurity series of welds GTA 13–15 was extended using a very high purity atmosphere where the oxygen level was below 1 wt.ppm. This weld, GTA 16, however exhibited a DBTT about the same as that of the intermediate purity weld. Further analysis revealed the presence of 63 wt.ppm (3200 at.ppm) hydrogen. The attempts to reduce the level of oxygen led to an increase in hydrogen. Although titanium is an excellent getter for oxygen in the range of 600–800°C, no hydride phase exists at reasonably attainable concentrations of hydrogen at these temperatures [7]. The result is reduction of water by the hot Ti and release of hydrogen into the welding glove box. Further research will be done with a hydrogen getter in addition to the heated Ti.

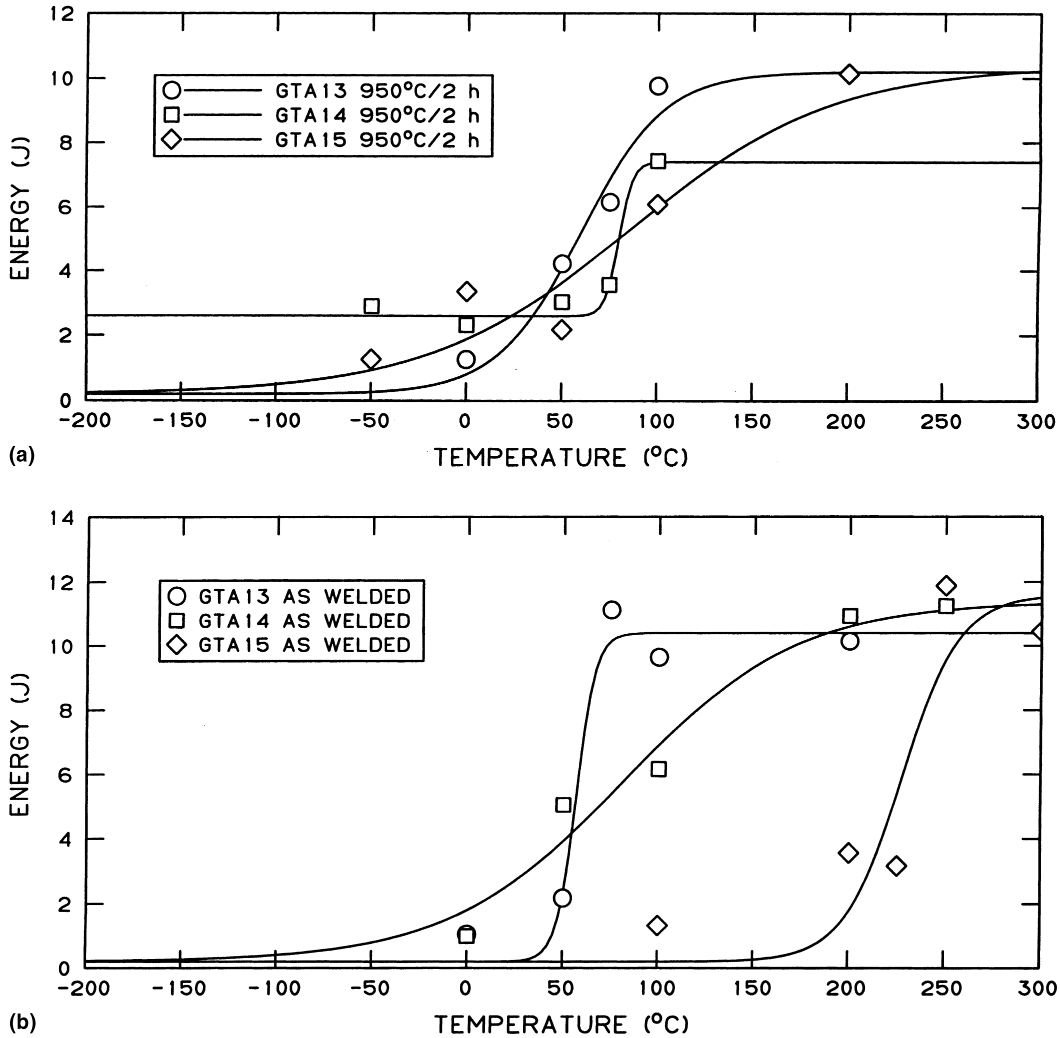

Fig. 5. Charpy impact curves for GTA welds of V–4Cr–4Ti of the impurity series. (a) As welded. (b) Following a post-weld heat treatment of 950°C/2 h.

Weld GTA 16 was out-gassed at 400°C for one hour to remove hydrogen [8]. Following this treatment, the DBTT was reduced by about 50°C. This is likely to be the result of removal of the hardening effect of hydrogen in solution [9]. The primary detrimental effect of hydrogen is not hardening in solid solution but rather embrittlement from formation of hydride. Embrittlement from hydride formation is believed to be the cause of the post-weld cracking that led to erratic Charpy test results. In the absence of a stress, hydride would not be expected to form above the solvus temperature of −65°C for 3000 at.ppm H [10]. However, a stress would be expected to shift the solvus temperature higher [11]. For the initial formation of hydride at a crack tip, the shift in temperature might be large since dislocations must be punched out at the interface with the hydride, not merely moved, so that stresses higher than the yield stress can exist locally. It has also been found that elevated oxygen levels will act synergistically with hydrogen to produce more severe embrittlement than either gas alone [12]. The strengthening effect of oxygen in solution might permit a higher stress at crack tips and thus a greater shift in the hydrogen solvus temperature. Westlake and Ockers confirmed the effect of oxygen on the solubility of hydrogen in vanadium and determined that the shift, about 25°C for concentrations in the present study, was not large in the absence of an external stress [13]. However, oxygen, even at the levels present in the weld samples, could make a measurable contribution in the case of an externally applied stress. Another explanation might be that the hydrogen and oxygen form water vapor in microvoids at the crack tip or at grain boundaries, causing pressurized voids that aid fracture, as observed in copper [14]. A specimen containing 850 wt.ppm oxygen in solution and doped with 2.5 at.% hydrogen was cooled to −45°C in a transmission electron microscope. No voids or bubbles could be found, and no diffraction patterns from ice were observed, but their observation would have been very difficult even if they were present. The conclusion is that hydrogen can have detrimental effects on welds in vanadium and must be eliminated from the welding atmosphere.

4. Conclusions

GTA welds were made in 6.4 mm plates of V–Cr–Ti alloys with a DBTT of 50°C. This DBTT could be attained either by making a high purity weld or by using a post-weld heat treatment. Although the PWHT is an important step in the welding process at the present time, it is speculated that the resultant grain boundary precipitation resulting from heat treating a weld with higher levels of impurities than experienced in this study might limit its usefulness. Therefore, atmospheric purity remains an essential factor in GTA welding of vanadium alloys.

The GTA welds have DBTTs about 150°C above similar EB welds. Control of interstitials is the most important factor in reducing the DBTT of GTA welds. However, other factors such as grain size, which has been demonstrated to influence mechanical properties in electron beam welds, will probably have to be controlled as well. Addition of trace elements or introduction of fine dispersions to aid in grain size control will be the subject of future research. In the past few years, large increments in reduction of embrittlement have been made in vanadium alloy welds. It is reasonable to expect that as the physical metallurgy of vanadium alloy welds is studied and mechanisms of embrittlement are discovered, progress will continue in vanadium alloy welding.

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