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# Effects of post-weld heat treatment conditions on hardness, microstructures and impact properties of vanadium alloys

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#### Abstract

Gas-tungsten-arc (GTA) weld joints were made from V–4Cr–4Ti alloys in which oxygen levels at the weld metal ranged from 73 to 355 wppm. Both the weld metal and the base metal of the joints showed hardening after the welding. In this study, the influence of post-weld heat treatment (PWHT) on hardness, microstructure and impact properties were investigated. Change in hardness due to annealing at 673 and 973–1273 K was shown to be caused by release of contaminant hydrogen and precipitate evolution, respectively. Results indicated that the precipitation hardening increased with oxygen level. Charpy impact properties did not change significantly due to annealing at 673 K, while fine precipitation at 1073 K degraded the impact property. No improvement of impact property by PWHT is expected at low oxygen level in the weld metal. Precipitation behavior during aging and irradiation may determine the performance of V–4Cr–4Ti weld joints.

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# 1. Introduction

Welding is a key technology for the low-activation vanadium alloys to fabricate a large component for fusion reactors. Weldability under gas-tungsten-arc (GTA), laser and electron-beam welding are investigated [1–3]. GTA weld joints produced from NIFS-HEAT-1, a Japanese reference V–4Cr–4Ti alloy with low oxygen level [4,5], exhibited better impact properties than similar weld joints produced from the US large heat [3]. The results indicated that the impact property significantly improved by reducing solute oxygen content in the weld metal. Post-weld heat treatment (PWHT) is known to improve the impact property of the weld joint. Previous study on the GTA welded V–4Cr–4Ti alloy has shown that the improvement in the property by PWHT at 1223 K was significant when ductile–brittle transition

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temperature (DBTT) before PWHT was 501 K, but small when it was 330 or 355 K [6]. Precipitation was reported as a possible cause of the property change by PWHT [6]. Systematic study of hardness and microstructural evolution with the heat treatment is necessary to investigate mechanism of the PWHT effect. The purpose of this study is to estimate PWHT effects on hardness, microstructure and impact property of the weld metal which may be useful for the mechanistic understanding.

## 2. Experimental procedure

GTA weld joints were made from the V-4Cr-4Ti alloy products, which were designated as NIFS-HEAT-1 (NH1) [4,5], HP [3] and US832665 (US) [7] by 6-pass welds. The products, welding procedure and conditions have been reported elsewhere [3]. Impurity levels of the weld joints are shown in Table 1. JGTA4 was additionally made by the same procedure as that for the other welds and therefore JGTA4 is expected to be

Table 1 Impurity levels of GTA weld joints of V–4Cr–4Ti alloys (wppm)

Plate/filler	Weld ID	Weld metal				Base metal				Condition
		С	Ν	0	Н	С	Ν	0	Н	
NH1/HP	JGTA2	70	97	73	69	60	100	189	86	As-welded
US/HP	JGTA3	80		135	59	120	71	338	51	As-welded
NH1/NH1	JGTA1	70	108	198	49	64	105	193	58 1	As-welded PWHT: 673 K × 1 h
	JGTA4	74	84	210	47	81	116	218	45	As-welded
US/US	GTA23		73	355	43	127	57	345	53	As-welded

NH1, US and HP refer to NIFS-HEAT-1 [4,5], US heat 832665 [7] and laboratory-melt high-purity ternary [3], respectively.

a duplicate of JGTA1. Oxygen level in weld metal varied with combination of the plate and the filler wire. All the weld joints were contaminated with hydrogen during GTA welding. PWHT was made at 473–1273 K for 1 h. As shown in Table 1, hydrogen concentration was reduced from 58 to 1 wppm by PWHT at 673 K. Distribution of Vickers hardness around weld metal was measured on JGTA1, JGTA2, JGTA3 and GTA23 after PWHT with the load of 500 gf for 30 s. Transmission electron microscopy (TEM) was conducted at the center of weld metal of JGTA1 and JGTA4. Charpy impact test with 1/3 size specimens  $(3.3 \times 3.3 \times 25.4 \text{ mm}^3)$  was performed using JGTA4 after PWHT at 673, 1073 and 1173 K. The V-notch on the Charpy specimens was 30° in included angle and 0.66 mm in depth.

## 3. Results

Fig. 1 shows change in hardness of the weld metal and the base metal of the weld joints after PWHT at various temperature for 1 h. Hardness level of NH1



Fig. 1. Change in hardness of the weld metal (WM) and the base metal (BM) after PWHT at various temperatures for 1 h.

plates and US plates before welds is indicated by a dark zone. Both the weld metal and the base metal was hardened by GTA welding. The hardening of the base metal by GTA welding occurred in all the weld joints at any place examined. After PWHT at 673 K both the weld metal and the base metal were softened. Hardness of the base metal recovered to the level before welding. Beyond 673 K, hardness of the base metal was almost constant. On the contrary, the weld metal exhibited hardening again after PWHT at 973 or 1073 K. After the peak the weld metal was recovered in hardness to the level before welding by PWHT at 1073–1273 K. The hardness of the weld metal increased with increasing oxygen level.

Fig. 2 shows TEM microstructures of the weld metal of NH1/NH1 welds. Before welding, two types of precipitates have been identified as Ti-rich and Ti-C-O compounds in NH1 plate [8]. In the as-welded condition, no precipitate but low density of dislocations  $(1.1 \times 10^{13})$ m<sup>-2</sup>) was observed. After PWHT at 873 and 973 K, TEM microstructures were similar to that in the aswelded condition. On the other hand, a high-density of fine precipitates oriented in (001) directions, were observed after PWHT at 1073 K. The precipitates were too thin to determine the composition with TEM-EDS (energy dispersive X-ray) analysis. The size of the precipitates increased, and the number density decreased with increasing PWHT temperature up to 1273 K. The average length and the number density of the precipitates are indicated below the TEM images.

Fig. 3 shows the results of Charpy impact test after PWHT at 673, 1073 and 1173 K for 1 h. DBTT was determined as the temperature where absorbed energy was 6.5 J, which is one half of the upper shelf energy  $(E_u = 13 \text{ J})$ . DBTT of NH1/NH1 welds in the as-welded condition was 188 K, which is 132 K lower than 320 K of US/US welds. After PWHT at 673 K, DBTT of NH1/ NH1 welds was 202 K, which is similar to that in the aswelded condition. PWHT at 1073 K, where the hardness



Fig. 2. TEM images of the NH1 plate before welds and the weld metal of NH1/NH1 weld joints before and after PWHT at 873–1273 K for 1 h.  $L_p$  and  $N_p$  are the average length and the number density of precipitates, respectively.



Fig. 3. Impact property of NH1/NH1 welds after PWHT at 673, 1073 and 1173 K for 1 h, compared with that of the as-welded and US/US welds.

peak occurred, raised the DBTT to 277 K. Because of insufficient data points, DBTT cannot be estimated after PWHT at 1173 K. However, the impact property does not seem to recover to the level in the as-welded condition.

Effects of oxygen level in weld metal and PWHT on DBTT are summarized in Fig. 4, which also includes previous data on US/US weld joints. Fig. 4 does not include the recent data on laser welding by Yan et al. [9] because welding method and Charpy specimen orientation were different from that in the present paper. The error bar indicated in Fig. 4 for the as-welded joints



Fig. 4. Correlation between oxygen concentration and DBTT of Charpy impact test. Data on previous study are included [3,6,10]. The arrow with the solid line means DBTT after PWHT at 1173 K and is estimated to be higher than 222 K. The other arrow with the dashed line indicates the recovery of DBTT by PWHT at 1223 K in US/US weld joint with 412 wppm oxygen. The error bar connects adjacent two test temperatures below and above the estimated DBTT.

connects adjacent two test temperatures below and above the estimated DBTT [3]. Since the Charpy tests were conducted every 50 K in most cases, DBTT has an error of 50 K. The solid line is a regression line for the as-welded data while the dashed line is for the previous data [6,10]. As indicated by an arrow on the dashed line, DBTT improved significantly when DBTT and oxygen level in the as-welded condition were 501 K and 412 wppm, respectively.

#### 4. Discussion

#### 4.1. Effect of PWHT on hardness

As shown in Fig. 1, the weld metal was harder than the base metal in all the weld joints. From the TEM results shown in Fig. 2 it is clear that the precipitates observed before welds dissolved during the welding. Therefore interstitial impurities, such as carbon, nitrogen and oxygen, were expected to be released into matrix and to cause solid solution hardening of the weld metal.

All the weld joints exhibited relatively large hardness recovery after PWHT at 673 K. Hardness of the base metal was recovered to the level of before welds after the PWHT at 673 K. Because no significant changes in dislocation structure was observed by the PWHT, the hardness recovery is considered to correspond to the release of hydrogen as shown in Table 1.

In Fig. 5, dependence of hardness for the weld metal on oxygen content is compared with that for the oxygendoped V-4Cr-4Ti model alloys in as-melted condition [8]. Since the slope of the two lines for the weld metal are close to that of the dashed line for the model alloys, variation in hardness of the weld metal may be explained in terms of solid solution hardening due to oxygen. The shift indicated by the arrow is attributed to the release of hydrogen. Possible reason of the gap between the model alloys and the weld metal after PWHT at 673 K is the difference in density of defects formed during solidification, or the difference in hydrogen concentration.

In NH1/NH1 welds, a close correlation was found between the hardness peak and appearance of the fine precipitates at 1073 K. This suggests precipitation



Fig. 5. Dependence of hardness of the weld metal before/after PWHT at 673 K on oxygen concentration. Hardness of the asmelted model alloys doped with oxygen is also plotted [8]. The arrow indicates hardness shift caused by degassing of hydrogen.

hardening around the hardness peak. The increase in the peak hardness at 973–1073 K with oxygen level is consistent with those for the model V–4Cr–4Ti alloys doped with oxygen [8].

### 4.2. Effect of PWHT on Charpy impact property

As shown in Fig. 3, DBTT was 202 K for NH1/NH1 after PWHT at 673 K, which is close to 188 K for the aswelded condition, implying that about 50 wppm of hydrogen did not affect the impact properties. On the contrary, for the US/US welds, PWHT at 673 K has improved DBTT by 20 or 47 K compared to that in the as-welded condition [10]. Hydrogen promotes hardening and ductility loss, the extent of which, however, has been revealed to depend on the oxygen level [11,12]. In the case of NH1/NH1 welds, hydrogen embrittlement may be reduced compared to US/US welds because of the reduced oxygen.

In Fig. 4 the slope of the dashed line was much higher than that of the solid line. The change in the dependence is possibly caused by precipitates, which were observed in the matrix of weld metal of US/US joint with 412 wppm oxygen in the as-welded condition [6]. No precipitates were observed in NH1/NH1 joint in this condition. The PWHT at 1223 K was very effective in improving the DBTT of the joint with 412 wppm oxygen, because the PWHT is expected to coarsen the precipitates and to reduce hardening by the precipitates. In the case of joints containing less than 350 wppm of oxygen, however, the improvement in DBTT by PWHT at higher temperatures is not expected because of the absence of precipitates in the as-welded condition. Instead, PWHT at 1073 K caused precipitation dispersion hardening and resulted in an increase in DBTT by about 80 K. DBTT did not recover after PWHT at 1173 K. The impact property of the weld metal can be more sensitive to PWHT than to oxygen content if PWHT induces precipitation.

No improvement of impact property by PWHT is expected at low oxygen level in the weld metal. On the other hand, precipitation behavior during the long-time aging and irradiation is expected to determine the performance of V-4Cr-4Ti weld joints, and remains to be studied.

# 5. Conclusions

Hardness recovery process, microstructure development and their effect on impact properties were investigated after the heat treatment of the weld joints of V-4Cr-4Ti alloys with various oxygen contents.

 Up to 673 K: hardness recovery occurred by release of hydrogen in both the weld metal and the base metal. Hardness of the base metal recovered to the level before welding. Charpy impact property did not change due to degassing.

- (2) 773–873 K: hardness remained constant. No microstructural change was observed.
- (3) 973–1073 K: hardness peak occurred and corresponded to fine precipitation. Charpy impact property was found to be degraded by the precipitation.
- (4) 1173–1273 K: hardness decreased due to coarsening of the precipitates. Hardness of the weld metal recovered to the level before welding.
- (5) The hardness of the weld metal was found to increase with oxygen level.
- (6) No improvement in impact property by PWHT is expected at low oxygen levels in the weld metal.

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