

# Effect of post-weld heat treatment on microstructure, hardness and low-temperature impact toughness of electron beam welds of NIFS-HEAT-2 and CEA-J57 heats of V–4Ti–4Cr alloy



V. Tsisar<sup>a,\*</sup>, T. Nagasaka<sup>b</sup>, M. Le Flem<sup>c</sup>, O. Yeliseyeva<sup>d</sup>, J. Konys<sup>a</sup>, T. Muroga<sup>b</sup>

<sup>a</sup> Karlsruhe Institute of Technology (KIT), Institute for Applied Materials – Applied Materials Physics (IAM-AWP), Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany

<sup>b</sup> National Institute for Fusion Science (NIFS), 322-6 Oroshi, Toki, Gifu 509-5292, Japan

<sup>c</sup> CEA Saclay, DEN/DMN/SRMA/LA2M, 91191 Gif Sur Yvette, France

<sup>d</sup> Physical-Mechanical Institute of National Academy of Sciences of Ukraine (PhMI NASU), 5 Naukova St., 79601 Lviv, Ukraine

## ARTICLE INFO

### Article history:

Received 13 November 2015

Revised 29 March 2016

Accepted 4 April 2016

Available online 5 May 2016

### Keywords:

V–4Ti–4Cr alloy

Electron beam welding

Microstructure

Hardness

Impact toughness

## ABSTRACT

Bead-on-plate electron beam welding in high vacuum atmosphere was applied to the plates of NIFS-HEAT-2 and CEA-J57 heats of V–4Ti–4Cr alloy. Effect of post-weld heat treatment (PWHT) in the temperature range 673–1273 K on the hardness, impact toughness at 77 K and microstructure of weld metal was investigated. After PWHT at 773 K, hardness of weld metal slightly decreases from 180 HV<sub>100</sub> (as-welded state) to ~170 HV<sub>100</sub> while absorbed energy increases up to ~10 J showing ductile fracture mode. PWHT at 973 K results in re-hardening of weld metal up to ~180 HV<sub>100</sub> caused by re-precipitation of Ti–C,O,N precipitates and corresponding decreasing absorbed energy to ~2 J with brittle fracture mode. PWHT in-between 1073–1273 K results in gradual recovery of hardness towards values comparable with those of base metal. Impact toughness (77 K) of weld metal after PWHT at 1073 K is not recovered neither to the value in as-welded state nor to that one of base metal.

© 2016 The Authors. Published by Elsevier Ltd.

This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

## 1. Introduction

V–4Ti–4Cr alloy is a candidate structural material for V/Li blanket of fusion reactor [1]. Application of V–4Ti–4Cr requires development of joining techniques. Gas-tungsten-arc (GTA) welding, as the most industrially developed method, as well as laser and electron beam (EB) welding techniques are used for joining vanadium alloys [2–7]. Because mechanical properties of vanadium alloys are very sensitive to the redistribution of precipitations (Ti–C,O,N) and non-metallic impurities [8–10], an additional pick-up of latter from the surrounding gas atmosphere should be minimized using during welding high-purity inert gases and filler wire [11]. Even if latter is provided, weld metal (WM) of vanadium alloys usually shows increase in hardness in comparison with base metal (BM) which is caused by solid-solution hardening of matrix by means of oxygen released in fusion zone from Ti–C,O,N precipitates, naturally present in V–4Ti–4Cr alloys [12]. Comparing Charpy impact energies of WM produced by gas-tungsten-arc (GTA), laser

and electron beam (EB) welding techniques for the same V–4Ti–4Cr alloy NIFS-HEAT-2 [6,7,13], it was shown that the GTA welding results in marked shift of ductile–brittle transition temperature (DBTT) to higher temperatures while impact properties of WM after laser welding remains similar to the BM [1]. EB welding provides even superior impact properties of WM, in comparison with BM and with WM obtained by means of GTA and laser welding techniques [1,6,7]. The marked shift in DBTT occurred for WM after the GTA welding was caused by additional pick up of oxygen from the gas atmosphere while oxygen content in WM after laser and EB welding was identical with that in BM indicating that high-vacuum atmosphere (EB welding) and high-purity argon (laser welding) mitigate additional contamination of vanadium alloys by interstitial impurities [1]. Because the oxygen content in WM after laser and EB welding should be similar, at least based on the similarity in hardness of WM (~180 HV), the superior impact properties of WM obtained by EB welding are attributed to the narrowest weld bead and fine-grained structure, in contrast to the wider weld beads and coarser structure formed in WM after laser and GTA welding [1]. It was concluded that PWHT is not necessary to be applied to the WM produced by laser and EB welding [6,7] while mechanical properties of WM produced by GTA welding

\* Corresponding author.

E-mail address: [valentyn.tsisar@kit.edu](mailto:valentyn.tsisar@kit.edu) (V. Tsisar).

**Table 1**

Chemical composition of V-alloys NH2 and J57 (mass%, \*wppm).

Heat	V	Cr	Ti	*C	*O	*N
NH2	Bal.	4.02	3.98	69	148	122
J57		3.76	3.93	70	290	110

could be recovered by means of PWHT [11]. However, a potential needlessness in PWHT for laser and EB welds does not exclude the necessity to study the effect of post-weld heating to working temperatures on the change of mechanical properties and structure of weld zones. It is important in the view of the temperature- and time-induced redistribution of precipitates and non-metallic impurities in the WM since re-precipitation-induced hardening affects substantially mechanical properties of vanadium alloys [8–11]. Application of PWHT becomes especially important in the light of the irradiation-induced hardening of weld metal (WM) in comparison with base metal (BM) taking place within the lower temperature range (~670–720 K) of fusion reactor blanket operation [14–16]. PWHT, it is believed, is a more appropriate solution than post-irradiation heat treatment (PIHT) in order to improve mechanical properties of WM [17]. Therefore, characterization of PWHT effect in un-irradiated state is of interest in order to distinguish among the hardening mechanisms and their contribution into the embrittlement of WM. In this work, the effect of PWHT on microstructure, hardness and low-temperature impact properties of EB welds of V–4Ti–4Cr alloy is investigated and discussed.

## 2. Material and methods

Two heats of V–4Ti–4Cr alloy, i.e., NIFS-HEAT-2 (NH2) and CEA-J57 (J57), were investigated in this work [18–20]. Table 1 shows chemical composition of NH2 and J57 [18,21]. EB welds were prepared by bead-on-plate welding in high vacuum atmosphere. Welding parameters are presented elsewhere [7]. Effect of welding on the chemical composition of WM with respect to non-metallic impurities was not investigated in this work. However, based on the results available from the laser welding for NH2, one can assume that oxygen content in WM after EB welding should be similar to that after laser welding, i.e. does not change in comparison with base metal indicating about absence of additional pick up of oxygen from the surrounding gas-atmosphere during welding [17].

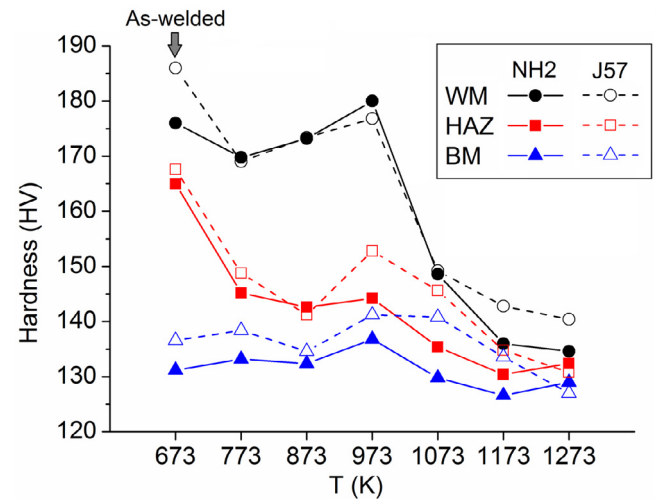
Charpy 1.3-size specimens ( $3.3 \times 3.3 \times 25.4 \text{ mm}^3$ ) were machined from the plates with weld bead. V-notch (0.66 mm in depth, angle of 30°, and root radius of 0.08 mm) is placed in the weld metal (WM) perpendicular to the rolling direction. Before PWHT, samples were placed in tantalum container which was wrapped by zirconium foil in order to provide additional gettering of vacuum atmosphere with respect to the non-metallic impurities during annealing. The PWHT was carried out in the temperature range 673–1273 K, in the high-vacuum atmosphere for 1 h.

After PWHT, impact toughness was determined at 77 K by means of drop weight impact tester. Vickers hardness of zones of weld joints was measured under the loading of 1000 gf ( $\text{HV}_{100}$ ) for 30 s. Light optical and scanning-electron microscopes were used in order to determine structural changes in zones of weld join and fracture mode depending on the temperature of PWHT.

## 3. Results and discussion

### 3.1. Hardness of weld zones after PWHT

In as-welded state followed by degassing at 673 K, the WM of both heats showed increase in hardness up to about 180  $\text{HV}_{100}$



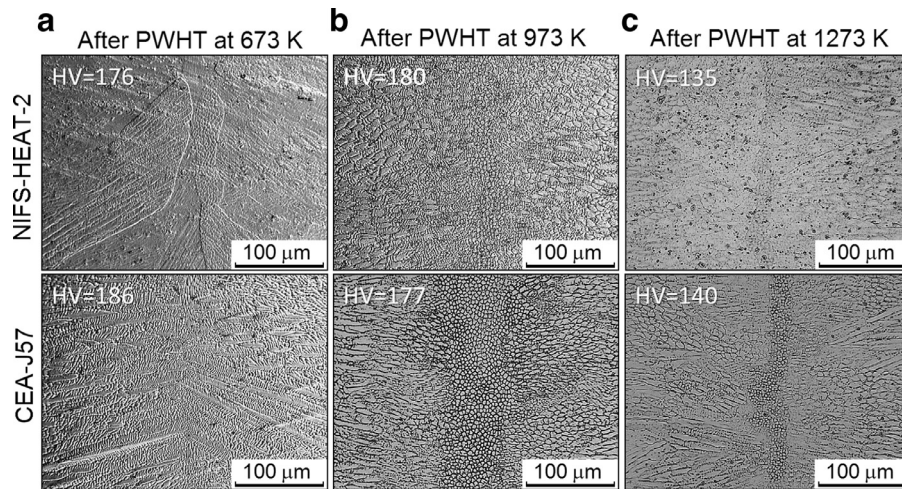
**Fig. 1.** Change in hardness ( $\text{HV}_{100}$ ) of weld metal (WM), heat affected zone (HAZ) and base metal (BM) of electron beam welds of NH2 and J57 heats of V–4Ti–4Cr alloy depending on the temperature of post-weld heat treatment.

in comparison with BM (~135  $\text{HV}_{100}$ ) caused by decomposition of Ti–C,O,N precipitates and subsequent solid-solution hardening of vanadium matrix by oxygen [7,22]. Hardness decreased gradually from WM (~1 mm thick) through HAZ (~2.5 mm thick from each side of bead center line) and further towards BM.

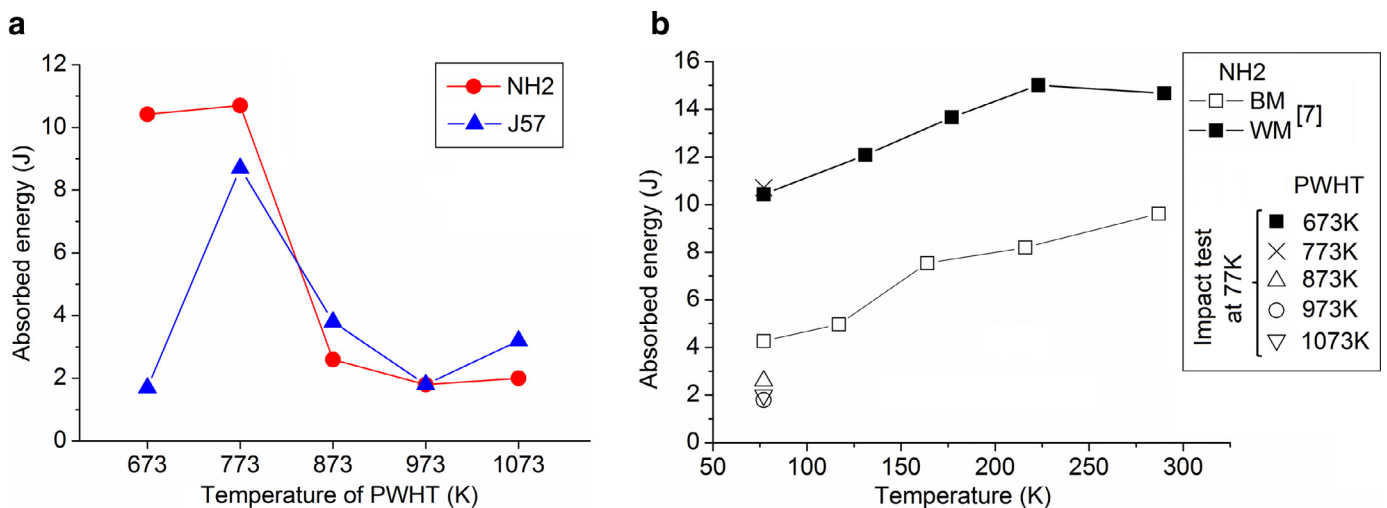
Fig. 1 shows change in hardness of WM and HAZ in comparison with BM depending on the temperature of PWHT. Both heats show in general quite similar behavior. Hardness profile of WM is also very similar to that one obtained after laser welding of NH2 [17]. This similarity is used hereafter for interpretation of phase-structural transformations in WM obtained by EB welding, based on the findings from transmission electron microscopy (TEM) for WM after laser welding [17].

After PWHT at 773 K, WM is slightly softened in comparison with as-welded state (Fig. 1). Softening of HAZ is more pronounced, i.e., hardness decreased from 165  $\text{HV}_{100}$ , in as-welded state, to 145  $\text{HV}_{100}$ , after PWHT at 773 K. A further increase in temperature of PWHT to 873 and 973 K results in re-hardening of WM to the level similar in as-welded state. Hardness in the HAZ, at the same time, increases noticeably only for J57, which initially contains more oxygen (290 mass ppm) in comparison with NH2 (148 mass ppm) (Table 1). It is supposed that re-hardening occurs due to the temperature-induced re-precipitation of Ti–C,O,N. This supposition is in a good agreement with literature data. Thus, similar peak in hardness at 973 K was observed by Heo et al. [9], which investigated effect of impurity levels on precipitation behavior of V–4Ti–4Cr alloys initially solution-annealed at 1373 K. Nishimura et al. [10], investigating similar effect on the mechanical properties of initially solution-annealed NH2, found that peak in hardness, yield and tensile strength appears at 973 K, as-well. Results obtained by Nagasaka et al. [11], also indicates about increase in hardness of WM, produced by GTA welding, after PWHT performed at 973 and 1073 K. In the case of laser welding, the peak in hardness for WM was observed after PWHT at 873 K [17]. Utilizing transmission electron microscopy (TEM) it was confirmed that re-hardening is caused by uniformly formed high-density and fine Ti–C,O,N precipitations which strengthening vanadium matrix [9–11,17].

Further increase in PWHT in-between 1073 and 1273 K results in gradual decrease in hardness of WM and HAZ to the values close to the BM (Fig. 1). It is caused by further oxygen gettering from the solid-solution to Ti–C,O,N precipitates and their subsequent coarsening. In contrast to the uniformly redistributed precipitates after



**Fig. 2.** Light optical micrographs of etched cross-sections of weld metal of electron beam welds of NH2 and J57 heats of V-4Ti-4Cr alloy after post-weld heat treatment at (a) 673, (b) 973 and (c) 1273 K.



**Fig. 3.** (a) Absorbed energy at 77 K of weld metal (WM) of electron beam welds of NH2 and J57 heats of V-4Ti-4Cr alloy depending on the temperature of post-weld heat treatment (PWHT). (b) Absorbed energies weld metal (WM) and base metal (BM) of NH2 depending on temperature of Charpy impact test in comparison with absorbed energies of WM after PWHT followed by impact test at 77 K.

PWHT at 873 and 973 K, the precipitates in WM after PWHT at 1073 K form a concentrated region which is heterogeneously redistributed in vanadium matrix [17].

### 3.2. Light optical microstructure of weld metal after PWHT

Fig. 2 shows evolution of morphology of WM depending on PWHT. It is clear, that structure of WM underwent marked transformations after PWHT. Thus, a columnar dendrite crystallites, elongated from the center of weld joint towards direction of heat removal (Fig. 2a), after PWHT at 973 K transformed into fine equiaxial grains observed in the center of WM and elongated grains observed at the periphery (Fig. 2b) as a result of partial recrystallization and fragmentation of initial dendritic structure developed in as-welded state due to fast cooling (Fig. 2a). Fine-grained structure observed in WM might also partially favor the re-hardening in WM; however the precipitation-induced hardening is a main strengthening mechanism after PWHT at 973 K (Fig. 1). Further increase in PWHT temperature up to 1273 K does not affect substantially the morphology of WM on both heats (Fig. 2), although the decrease in hardness clearly indicates about

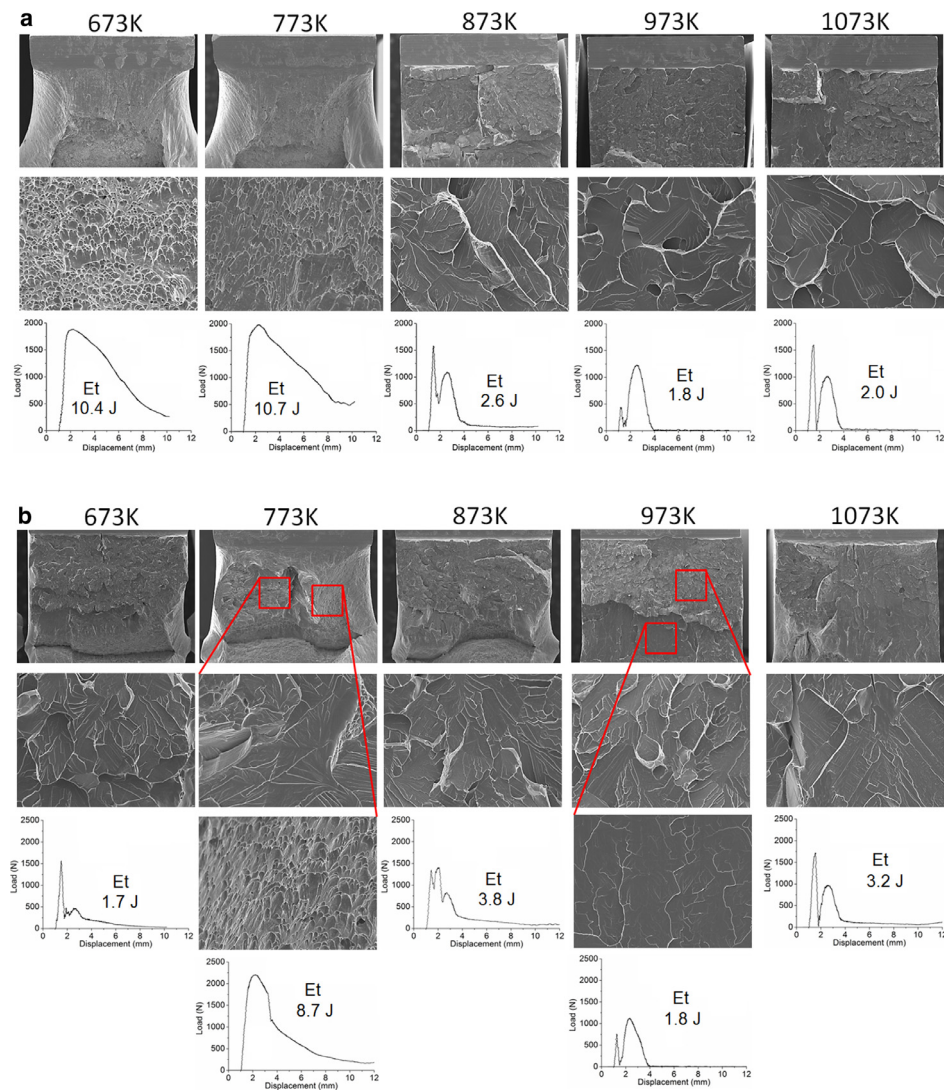
recovery of mechanical properties of WM to those which are peculiar for BM (Fig. 1).

PWHT does not affect the structure of HAZ, although the hardness markedly decreases with temperature (Fig. 1).

### 3.3. Impact toughness of weld metal at 77 K

Fig. 3a shows the results of Charpy impact test performed at 77 K on samples of EB weld joints after PWHT in-between 673–1073 K. PWHT at 773 K improves impact toughness of WM. Absorbed energies increase, in comparison with as-welded state, slightly from 10.4 to 10.7 J for NH2 and markedly from 1.7 to 8.7 J for J57 (Fig. 3a). Fracture mode of NH2, similar to the as-welded state, remains ductile that is reflected also on the load-displacement curve showing significant strain followed by gradual load decrease at fracture (Fig. 4a). In the case of J57, the brittle fracture, observed after impact test in as-welded state, changes into mixed mode, i.e. brittle-ductile (Fig. 4b). Thus, PWHT at 773 K does not affect substantially the impact properties of NH2 EB weld and improves markedly that of J57.

Ti-C,O,N precipitate-induced re-hardening of WM observed after PWHT at 873 K and especially at 973 K impairs impact



**Fig. 4.** Fracture surface of weld metal (WM) of electron beam welds of (a) NH2 and (b) J57 heats of V-4Ti-4Cr alloy depending on the temperature of post-weld heat treatment after Charpy impact test performed at  $\sim 77$  K.

properties of EB welds in comparison with as-welded state and that one after PWHT at 773 K. Thus, at the peak of hardness after PWHT at 973 K, the absorbed energies of both heats decrease to 1.8 J, that is even lower in comparison with BM (Fig. 3a and b). The repeated sharp drop in load on the load–displacement curve, as well as the failure by cleavage, indicate the brittle fracture mode (Fig. 4). Aging at 973 K for 100 h in liquid Li does not change low-temperature impact energy (2.2 J) and fracture mode of WM on NH2 [22]. Muroga et al., reported that for WM obtained on NH2 by laser welding, with the highest hardening level after PWHT at 873 K, the time required for recovering impact properties to the level comparable to that in as-welded state is about 3000 h at 873 K [17].

Further increase in PWHT to 1073 K does not affect substantially the low-temperature (77 K) impact properties of EB welds in spite of marked decrease in hardness that clearly testifies in the further redistribution of oxygen from the solid-solution towards precipitates (Fig. 1). Absorbed energies of WM are 2.0 and 3.2 J for NH2 and J57, respectively, while fracture mode is brittle (Fig. 3a and Fig. 4). Thus, for NH2, the absorbed energy of WM after PWHT at 1073 K does not recover to the level comparable with that in BM ( $\sim 4$  J) (Fig. 4b) with ductile fracture mode [7].

Charpy impact tests after PWHT at higher temperatures were not performed since it is known that at temperatures  $> 1273$  K the dissolution of Ti-C,O,N again takes place resulting in increase in hardness of V-matrix by released oxygen and subsequent degradation of mechanical properties [10,17,23]. It is interesting to notice, when comparing Charpy test results for NH2 in solution-annealed state at 1373 K, for WM in as-welded state after EB and laser welding, and BM after vacuum annealing at 1273 K, that higher DBTT is associated with 1373 K annealing, indicating that larger fraction of oxygen transfers into solid-solution after this treatment [6,7,10].

Based on the results of impact test at 77 K it is difficult to choose the best PWHT temperature which might improve the impact properties of WM at least to the level as in the BM. However, more completed results obtained for laser welding of NH2, suggest that PWHT at 1073 K effectively recovers hardness and impact properties [17].

The present results indicate that the brittleness of EB welds of vanadium alloys is affected, expectedly, by the structure of WM, level of oxygen in solid-solution and precipitates. In the as-welded state, the structure of WM predetermines superior impact properties, i.e., overrides the negative influence of oxygen-induced

solid-solution hardening. The same level of hardening, which however induced by means of Ti–C,O,N precipitates, in contrast, diminishes positive effect of fine-grained structure of WM observed after PWHT at 973 K. PWHT at 1073 K should result in recovering of mechanical properties of WM due to further consumption of oxygen from the solid-solution and further coarsening of precipitations.

#### 4. Conclusions

An effect of post-weld heat treatment (PWHT) on the hardness, microstructure and low-temperature (77 K) impact properties of electron beam welds of NIFS-HEAT-2 and CEA-J57 heats of V–4Ti–4Cr alloy was investigated. It is determined that PWHT affects substantially morphology, hardness and impact toughness at 77 K of weld metal. Weld zones formed on the both heats showed similar properties after PWHT performed in-between 773 and 1273 K. The conclusions are as follows:

- PWHT performed at 773 K, improves impact toughness of weld metal at 77 K in comparison with as-welded state, that is reflected in higher absorbed energy ( $\sim 10$  J) ductile fracture mode and slight decrease in hardness ( $\sim 170$  HV);
- PWHT at 873 and 973 K causes Ti–C,O,N precipitation-induced re-hardening up to  $\sim 180$  HV resulting in degradation of impact properties of weld metal at 77 K in comparison with as-welded state and base metal;
- PWHT in-between 1073 and 1273 K results in gradual recovery of hardness of WM due to oxygen trapping from the solid-solution into Ti–C,O,N precipitates and their subsequent coarsening;
- Absorbed energy (impact test at 77 K) of weld metal of NIFS-HEAT-2 after PWHT at 1073 K (2.0 J) does not recover to level in as-welded state (9.5 J) and base-metal (4.3 J) in spite of marked hardness recovery from  $\sim 180$  to 150 HV;
- Absorbed energy (impact test at 77 K) of weld metal of CEA-J57 after PWHT at 1073 K recovers to the higher level (3.2 J) than in as-welded state (1.7 J);
- Heating of electron beam welds of V–4Ti–4Cr alloy to the working temperatures of blanket of fusion reactor, i.e.  $\sim 973$  K, embrittles the weld metal. In order to avoid precipitates-

induced embrittlement of weld metal, the PWHT at temperature  $\sim 1073$  K should be performed even in spite of the fact that impact properties of weld metal in as-welded state are superior in comparison with base metal.

#### References

- [1] T. Muroga, J.M. Chen, V.M. Chernov, R.J. Kurtz, M. Le Flem, J. Nucl. Mater. 455 (2014) 263–268.
- [2] M.L. Grossbeck, J.F. King, D.J. Alexander, P.M. Rice, G.M. Goodwin, J. Nucl. Mater. 258–263 (1998) 1369–1374.
- [3] M. Grossbeck, J. King, D. Hoelzer, J. Nucl. Mater. 283–287 (2000) 1356–1360.
- [4] M.L. Grossbeck, J.F. King, T. Nagasaka, S.A. David, J. Nucl. Mater. 307–311 (2002) 1590–1594.
- [5] H. Chung, J.-H. Park, R. Strain, K. Leong, D. Smith, J. Nucl. Mater. 258–263 (1998) 1451–1457.
- [6] N. Heo, T. Nagasaka, T. Muroga, A. Nishimura, K. Shinozaki, N. Takeshita, Fusion Eng. Des. 61–62 (2002) 749–755.
- [7] V. Tsisar, T. Nagasaka, M. Le Flem, O. Yeliseyeva, J. Konys, T. Muroga, Fusion Eng. Des. 89 (2014) 1633–1636.
- [8] K. Natesan, J. Nucl. Mater. 115 (1983) 251–262.
- [9] N.J. Heo, T. Nagasaka, T. Muroga, H. Matsui, J. Nucl. Mater. 307–311 (2002) 620–624.
- [10] A. Nishimura, A. Iwahori, N. Heo, T. Nagasaka, T. Muroga, S.-I. Tanaka, J. Nucl. Mater. 329–333 (2004) 438–441.
- [11] T. Nagasaka, T. Muroga, M. Grossbeck, T. Yamamoto, J. Nucl. Mater. 307–311 (2002) 1595–1599.
- [12] T. Nagasaka, T. Muroga, K.-i. Fukumoto, H. Watanabe, M.L. Grossbeck, J. Chen, Nucl. Fusion 46 (2006) 618–625.
- [13] T. Nagasaka, J.F. King, M.L. Grossbeck, T. Muroga, Fusion Technol. 39 (2001) 664–668.
- [14] H. Watanabe, N. Yoshida, T. Nagasaka, T. Muroga, J. Nucl. Mater. 417 (2011) 319–322.
- [15] T. Nagasaka, T. Muroga, H. Watanabe, T. Miyazawa, M. Yamazaki, K. Shinozaki, J. Nucl. Mater. 442 (2013) S364.
- [16] T. Nagasaka, N.-J. Heo, T. Muroga, A. Nishimura, H. Watanabe, M. Narui, K. Shinozaki, J. Nucl. Mater. 329–333 (2004) 1539–1543.
- [17] Takeo Muroga, Nam-jin Heo, Takuya Nagasaka, Hideo Watanabe, Arata Nishimura, Kenji Shinozaki, Plasma Fusion Res. 10 (2015) 1405092-1–1405092-7.
- [18] V. Duquesnes, T. Guilbert, M. Le Flem, J. Nucl. Mater. 426 (2012) 96–101.
- [19] M. Le Flem, J.-M. Gentzittel, P. Wident, J. Nucl. Mater. 442 (2013) S325.
- [20] T. Nagasaka, N. Heo, T. Muroga, M. Imamura, Fusion Eng. Des. 61–62 (2002) 757–762.
- [21] T. Muroga, T. Nagasaka, K. Abe, V.M. Chernov, H. Matsui, D.L. Smith, Z.-Y. Xu, S.J. Zinkle, J. Nucl. Mater. 307–311 (2002) 547–554.
- [22] V. Tsisar, T. Nagasaka, T. Muroga, T. Miyazawa, O. Yeliseyeva, J. Nucl. Mater. 442 (2013) 528–532.
- [23] N.J. Heo, T. Nagasaka, T. Muroga, J. Nucl. Mater. 325 (2004) 53–60.