Heterogeneous Precipitation and Mechanical Property Change by Heat Treatments for the Laser Weldments of V-4Cr-4Ti Alloy

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Bead-on-plate welds were produced on the 4 mm-thick V-4Cr-4Ti alloy (NIFS-HEAT-2), using a 2.0 kW YAG laser. The post-weld heat treatments (PWHT) were carried out in various conditions. Microstructures, Vickers hardness and Charpy impact properties were obtained for the weld metal after the PWHT. After PWHT for one hour, the hardness increased and after the peak declined with temperature. At 873 K, the hardness increased and after the peak declined with the time of PWHT. Microstructural observation showed that high density of fine precipitates formed homogeneously when the hardness increased, but the precipitate distribution changed into heterogeneous forming islands of developed precipitate aggregates, coincident with decrease in hardness and recovery of impact properties. Optical microscope observations suggested that a cellular structure of precipitate aggregate region was formed by PWHT. Microchemical analysis showed that Ti was enriched in the precipitate aggregate region. Therefore the areal oscillation of Ti concentration with cellular structures formed by melting and resolidification during the welding resulted in the heterogeneous precipitation by the following PWHT. The precipitation in the Ti-rich area purified the matrix of the weld metal and induced the recovery of hardening and impact property degradation. Optimum PWHT conditions were discussed according to the present results.

Keywords: vanadium alloy, laser welding, post-weld heat treatment, precipitation, hardness, Charpy impact property

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1. Introduction

Vanadium alloys, with a leading candidate of V-4Cr-4Ti, have been regarded as promising candidates for low activation blanket structural materials of fusion reactors. Recent efforts have made a significant progress in fabrication, joining and coating technology of the alloys [1,2]. Especially the welding technology has advanced largely including gas-tungsten-arc (GTA) welding [3,4], laser welding [5] and electron beam (EB) welding [6].

The laser welding has an advantage of field joining potential with simple apparatus. In the authors' previous study, technology for welding with suppressed pickup of environmental impurities has been enhanced by controlling the welding atmospheres, and the resulting joints of V-4Cr-4Ti showed good mechanical properties [7].

In spite of the good performance in as-weld conditions, degradation by irradiation are serious for the joints. Neutron irradiation at 563 K to 0.08 dpa already induced enhanced hardening of the weld metal and caused embrittlement at 77 K [8]. Neutron irradiation at 720 K to 5.3 dpa resulted in the ductile-brittle transition temperature (DBTT) exceeding 423 K, although the change in the base metal was much smaller keeping DBTT below RT [9]. The serious degradation of the weld joint by irradiation is considered to be due to enhanced radiation hardening. Welding causes evaporation of Ti-C, O, N precipitates, which otherwise capture C, O and N impurities in the matrix and enhance ductility of the alloy. Neutron irradiation induces formation of high density of fine precipitates or dislocations decorated with the precipitates accumulating impurities in the matrix, resulting in large hardening and embrittlement [10].

Possible improvement measures of the radiation performance of the joints include post-weld heat treatment (PWHT) and post-irradiation heat treatment (PIHT). In the case of irradiation at 720 K to 5.3 dpa, recovery of DBTT from > 423 K to 243 K took place by PIHT of 873 K and 1 h, coincident with partial recovery of dislocations and coarsening of precipitates [9]. Data for the effects of PWHT is, however, limited, although PWHT is more practical solution than PIHT considering difficulty in heating the blanket components during the interval of the reactor operation. An example of the limited efforts is that, by PWHT at 1223 K, radiation hardening at the weld metal was reduced to the level similar to that of the base

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Table 1 Impurity compositions of NIFS-HEAT-2 before and after welding and PWHT (wt.% for Cr and Ti and wppm for C, N, O).

		Cr	Ti	C	N	O
Before welding		4.12	4.13	51	126	139
After welding (weld metal)		-	-	40	139	138
PWHT 873K, 3000 h	Before PWHT (base metal)	-	-	58	133	179
	After PWHT (base metal)	-	-	78	123	181

metal [8].

Thus, optimization of the PWHT conditions before irradiation is necessary first of all. Some previous efforts showed heat treatment of weld joints recovered DBTT [3,5], but systematic investigation has not been carried out.

The purpose of this study is to clarify microstructure developments and changes in mechanical properties of V-4Cr-4Ti alloy weldments with various PWHT conditions, including long-term aging at the expected service temperature. The results will be discussed with respect to redistribution of impurities during the welding and the PWHT. An optimum PWHT condition will be discussed based on microstructures and mechanical properties.

2. Experimental Procedure

The material used in this study was 4 mm thick plate of NIFS-HEAT-2 (V-4Cr-4Ti) annealed at 1273 K for 2 hours, which was fabricated by the National Institute for Fusion Science [11]. The welds were prepared by bead-on-plate welding in a simple environmental control box, using 2.0 kW YAG laser. The welding apparatus is illustrated in details elsewhere [12, 13]. High-purity Ar gas was supplied to the control box to prevent the contamination with the interstitial impurities. In this study Ar flow rate for each nozzle was 150 L/min. For the welding, average power and welding speed were 1.6 kW and 0.33 m/min, respectively, laser focal point was on the plate surface, and the pulse duration and the frequency were 5 milliseconds and 100 Hz, respectively. Table 1 shows C/N/O level of the specimen before and after laser welding. The chemical analysis revealed that there was no significant pickup of the interstitials.

One-third V-notch Charpy (1/3 CVN) impact specimens were machined from the welds. Their dimensions were 3.3 mm × 3.3 mm in cross section and 25.4 mm in length. The notch was 0.66 mm in depth, and was machined from the weld metal for cracks to propagate parallel to the welding direction.

The PWHT was carried out with the specimens encapsuled into quartz glass to prevent the contamination with impurities. 1/3 CVN specimens, coupons for chemical analysis, and 0.25 mm and 1 mm thick plates including the welds prepared for microstructural observation and hardness measurement, respectively, were sandwiched between tantalum sheets and were wrapped in zirconium foil. The wrapped specimens were inserted in a quartz glass tube, which was evacuated to about 10^{-5} Pa and then was sealed. The specimens were then heat-treated at 873 K to 1223 K for one hour to investigate microstructural development and the impact property with the short-term PWHT. Isothermal heat treatment at 873 K was also carried out for one hour to 3000 hours. Table 1 also indicates the result of chemical analysis for the specimen heat treated at 873 K for 3000 hours showing the pickup of C/N/O impurities during the heat treatment was negligible.

After the PWHT, Charpy impact test was carried out from 77 K to room temperature. Vickers hardness measurement in the weld metal and the base metal with a load of 100 gf and a loading time of 30 seconds, and microstructural observations by optical microscope and TEM (Transmission Electron Microscope), including EDS (Energy-Dispersive X-ray Spectroscopy) analyses, were also carried out.

3. Results

3.1 Temperature dependence

Figure 1 indicates the change in Vickers hardness of the samples as a function of PWHT temperature from 673 K to 1373 K. Hardness of the as-welded was higher than that of the base metal by about 30 Hv. PWHT at 673 K did not cause significant change in hardness. The hardness increased, reached a peak at 873 K, and then gradually decreased to 1223 K with temperature. PWHT above 1223 K resulted in the increase in hardness again.

Figure 2 is a summary of the microstructural development according to PWHT at 873 K to 1373 K for one hour. At 873 K and 973 K, high density of fine precipitates were uniformly formed in the whole specimen. On the other hand, the precipitates in the specimen annealed at and above 1073 K were heterogeneously formed and were segregated. Especially, annealing at 1073 K caused tiny precipitates to form concentrated aggregate regions. At higher temperature, the regions were composed of lower density of coarsened precipitates. Lower pictures in Fig. 2 were taken in a lower magnification for the three high temperatures showing that the precipitate aggregates made island structures.

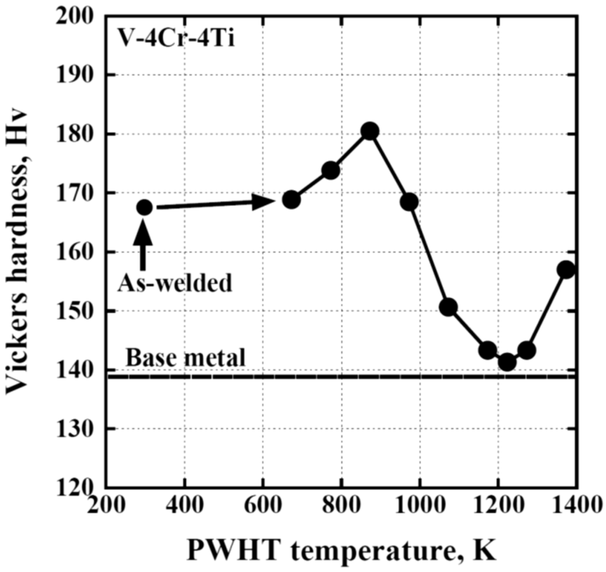

Fig. 1 Hardness change of the weld metal as a function of the PWHT temperature. The PWHT was carried out for one hour. A hardness peak was observed at 873 K.

Figure 3 shows the results of Charpy impact tests of the specimens with different PWHT temperature. While absorbed energy for PWHT at 973 K was lower than that for the as-welded, PWHT at 1073 K showed drastic increase in absorbed energy. The absorbed energy decreased again at 1223 K.

3.2 Time dependence

Figure 4 shows change in the hardness for the weld and base metal with the PWHT time of one hour to 3000 hours at 873 K. The hardness linearly increased to 10 hours, and then decreased. Significant change in hardness was not observed for the base metal by the PWHT.

Figure 5 illustrates development of the TEM microstructure of the weld metal during PWHT at 873 K. At one hour and 10 hours, high density of fine precipitates were observed homogeneously in the weld metal. The den-

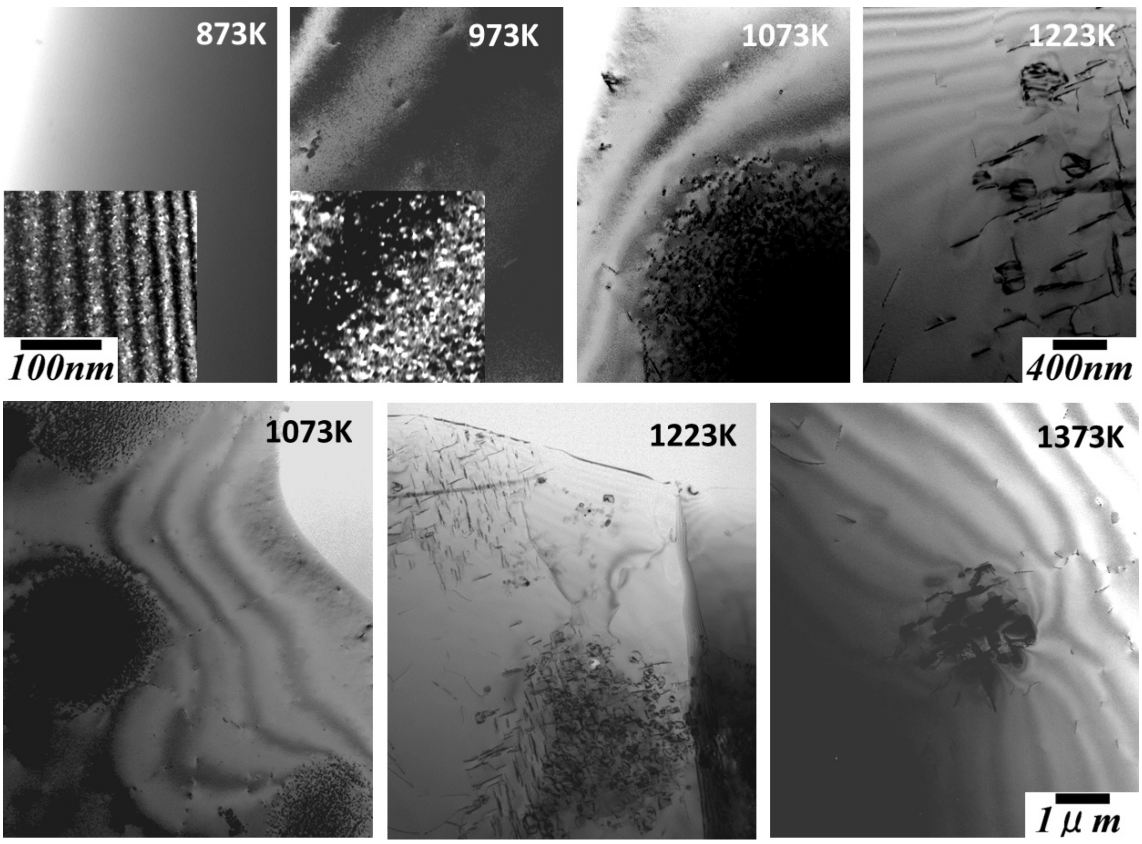

Fig. 2 Microstructural development of the weld metal with the PWHT temperature. At 873 K and 973 K, dark field images show tiny precipitates (white spots) were uniformly formed in the whole specimen. At 1073 K and above, the precipitates were formed heterogeneously. The lower figures taken in a lower magnification show precipitate aggregates formed islands at higher temperature.

sity of the dot images was highest at 10 hours. Heterogeneous precipitation was observed with the annealing time of 100 hours and longer. The lower figures are low magnification images at 100 hours and 3000 hours showing precipitate island structures. Figure 6 indicates Ti concentration profile measured by EDS, after PWHT at 873 K and 1000 hours, clearly showing Ti enrichment at the precipitate aggregate region.

Figure 7 shows optical micrographs of base metal, fusion line area and weld metal after PWHT at 873 K and 1000 hours. A cellular structure is observed, with a typical cell size of ~0.02 mm, in weld metal. The thick cell boundaries observed are considered to be representing the precipitate aggregate area. In the TEM observations, cross sectional views of the boundaries were obtained as islands of precipitate aggregates, as shown in Figs. 2 and 5 as a re-

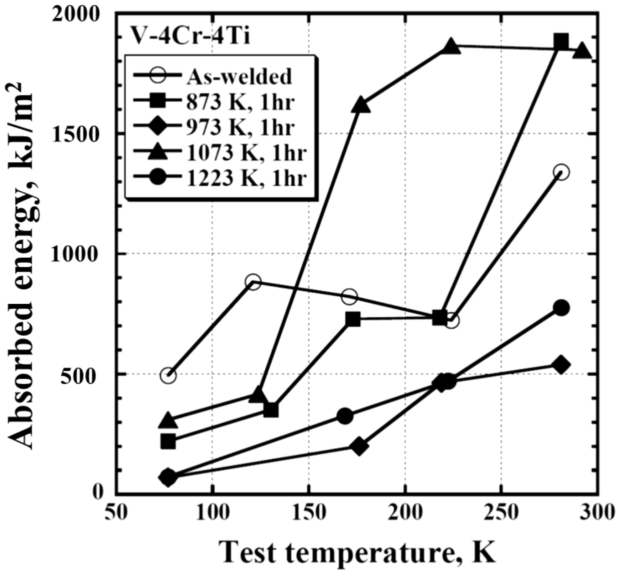

Fig. 3 Change in absorbed energy by the Charpy impact tests for the weld metal after PWHT for one hour at various temperature, as a function of test temperature.

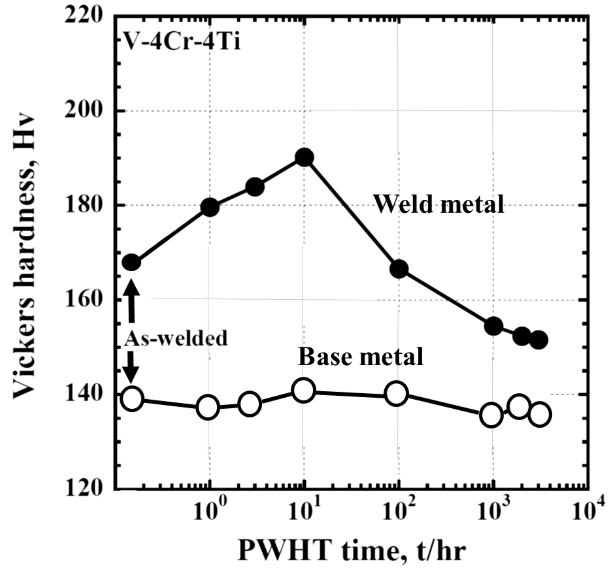

Fig. 4 Hardness change of the weld metal as a function of the time of PWHT at 873 K. A hardness peak was observed at 10 hours.

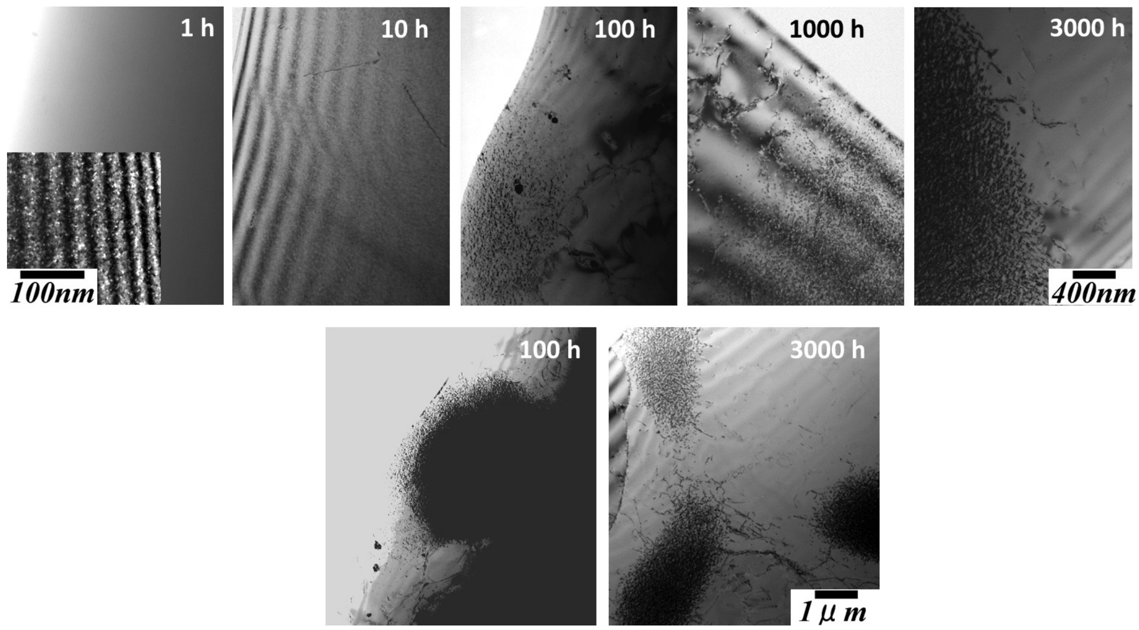

Fig. 5 Microstructural development of the weld metal with the time of PWHT at 873 K. High density of fine precipitates (white spots in dark field image) were observed uniformly in the whole specimens up to 10 hours. The heterogeneous precipitation occurred from 100 hours. The lower figures taken in a lower magnification show precipitate aggregates formed islands for longer PWHT time.

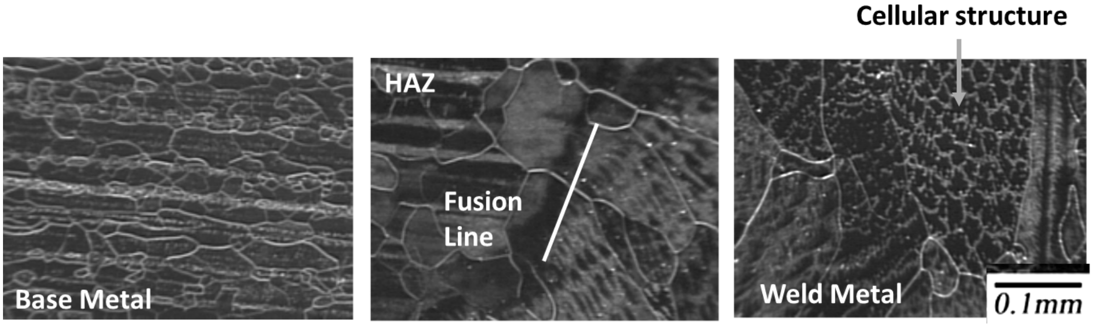

Fig. 7 Optical microstructure of base metal, fusion line area and weld metal after PWHT at 873 K and 1000 hours. Cellular structures with typical cell size of ~0.02 mm were observed in the weld metal.

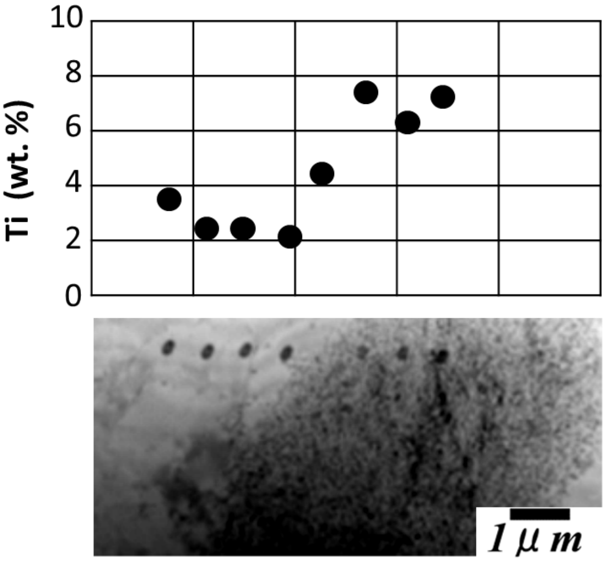

Fig. 6 Ti concentration profile including precipitate aggregate area detected after PWHT at 873 K and 1000 hours.

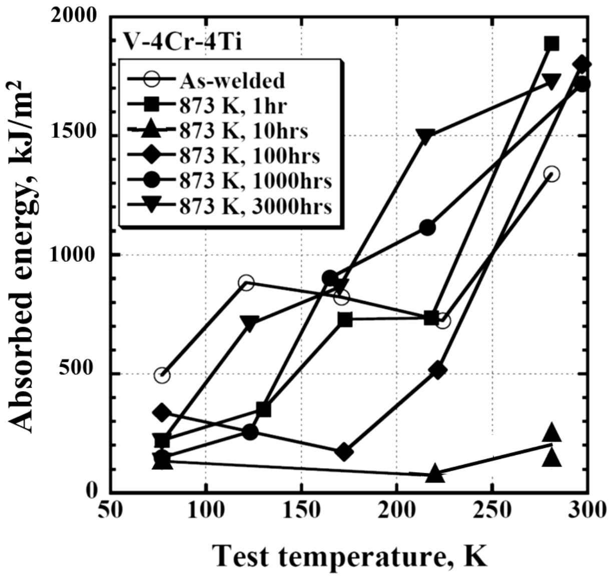

Fig. 8 Change in absorbed energy by the Charpy impact tests for the weld metal after PWHT at 873 K for various time, as a function of test temperature.

sult of slicing into thin foils prior to the TEM observations.

The result of Charpy impact tests of the specimens after PWHT at 873 K is plotted in Fig. 8. The absorbed energy after PWHT for one hour was comparable to that for the as-welded specimen at and above 173 K of the test temperature. However significant drop in the absorbed energy was observed after PWHT for 10 hours, showing absorbed energy lower than any other sample investigated. With PWHT for 100 hrs and above, the absorbed energy increased gradually with increasing the PWHT time, and was comparable to that of the as-welded specimen at 3000 hours.

4. Discussions

4.1 Effect of PWHT temperature

A previous study reported that larger Ti-rich and smaller Ti-C-O precipitates were formed in the base metal before laser welding. Both precipitates were dissolved in the matrix during the welding process [13]. The increase in hardness in as-welded condition, shown in Fig. 1, is explained by the increase in concentration of the interstitial impurity in solid solution in the matrix.

PWHT at 873 K and 973 K for one hour, by which high density of precipitates were formed as shown in Fig. 2, resulted in further increase in hardness. In these cases the absorbed energy decreased considerably. Thus, the high density of uniform precipitation has stronger effects on the mechanical properties than the solid solution state of the impurities does. It was shown that the drastic drop in the elongation and fracture strain properties and the increase in hardness and yield strength were attributed to the formation of fine precipitates, by the study of solution heat treatment followed by re-heating for precipitation [14]. However, it is interesting that there is a difference in temperature of the peak hardness (873 K) and the lowest absorbed energy (973 K), suggesting that the hardness obtained at RT cannot be the perfect index of the impact properties..

By PWHT at 1073 K and 1223 K, the precipitates aggregated non-uniformly in the area where Ti level is locally high. The structure of the precipitation aggregate regions formed a cellular structure, as indicated in Fig. 7. The increase in PWHT temperature caused an increase in size and decrease in the number density of the precipitates.

These results suggest that, with the increase in the PWHT temperature, precipitates evolved in Ti-rich area absorbing impurities in the surrounding region. The impurity level in the matrix thus became lower. The purification of the matrix by this process is considered to be the reason for the softening and the improvement of the impact properties of the joints shown in Figs. 1 and 3, respectively.

As to the degradation of the impact properties at 1223 K, the similar change was reported by the study of solution heat treatment followed by re-heating [14], in which degradation of impact properties occurred between 1173 K and 1273 K accompanied by increase in hardness. In that study it was deduced that the dissolution of the precipitate already started at this temperature although the observed microstructural change was small. Thus the degradation of the impact properties at 1223 K in the present study would be induced by partial dissolution of the precipitates increasing the impurity level in the matrix again.

4.2 Effect of PWHT time

The hardness kept increasing up to 10 hours of PWHT at 873 K as shown in Fig. 4, when the fine precipitates were distributed uniformly in the whole specimen. The absorbed energy dropped drastically by PWHT for 10 hours. These fine and uniform precipitates are responsible for the increase in hardness and the decrease in absorbed energy. As the PWHT time was increased to more than 100 hours, the precipitates formed aggregated regions with a cellular structure, and then hardness started to decrease. The cellular structure resulted in the reduction in the impurity concentrations of the matrix. The reduction in the impurity level is responsible for the significant decrease in hardness and the recovery of the impact property. These stories are identical to those of the PWHT temperature dependence. However, the second hardening and degradation of impact properties, as were seen at high temperature in Figs. 1 and 3, did not occur for the time dependence because precipitate dissolution did not take place at 873 K even when the PWHT time was very long.

4.3 Comparison with past research

Grossbeck *et al.* carried out PWHT at 1223 K and for 2 hours on GTA weldment of V-4Cr-4Ti, and showed that the effects of PWHT is limited [3]. In this case DBTT before and after PWHT were 330 - 501 K and 311 - 359 K, respectively. Because the oxygen level before and after PWHT ranges from 352 to 412 wppm, which is significantly higher than the present study, the effects of PWHT to reduce the matrix impurity level is thought to be limited. In addition, according to Fig. 3, the optimum PWHT temperature could be lower than 1223 K.

Chung *et al.* carried out PWHT at 1273 K and one hour on laser and EB welds of V-4Cr-4Ti, and found significant downward shift of DBTT [5]. They have examined microstructure after PWHT and concluded that dislocation recovery and precipitate aggregation contributed to cleaning the matrix. Their expression of the microstructure is consistent with that of the present study. But the present study showed that the cellular structure of Ti-rich area, typically with the cell size of ~0.02 mm, was already formed by the welding, enhancing the heterogeneous precipitation during PWHT.

4.4 PWHT optimization

It is difficult to recover the separation of Ti-rich and Ti-depleted area by PWHT. However, the separation may be thought to be preferable. Preferential precipitate evolution in Ti-rich area by PWHT, purifying Ti-depleted area, would enhance mechanical properties. Base on this concept, the suggested PWHT condition to form stable and heterogeneous precipitates and to purify the matrix would be 1073 K and one hour, within the present test matrix.

Since 873 K is the typical operation temperature of vanadium alloys as the structural materials of fusion blankets, Figs. 4, 5 and 8 are considered to indicate the aging effects of the weld joint during operation when PWHT is not applied. They suggest mechanical properties degrade in very early period of the operation, but then recover. An appropriate PWHT can avoid the degradation.

5. Conclusions

The microstructural developments and change in the mechanical properties of the laser weldments of a V-4Cr-4Ti alloy were investigated in various PWHT conditions.

1) PWHT for one hour at 873 K and 973 K resulted in the decrease in the absorbed energy. The hardening due to formation and uniform distribution of fine precipitates is responsible for the degradation of the impact property.

2) At 1073 K for one hour, cellular structures of the region, where Ti was locally enriched and precipitates aggregated, were formed. The hardening and the degraded impact properties recovered accordingly. At higher temperature, similar cellular structures were formed but the impact properties degraded again because partial dissolution of the precipitates and resulting impurity resolution occurred.

3) With the time of PWHT at 873 K, hardness increased to 10 hours and then decreased, coinciding with degradation and recovery of the impact properties. Precipitate aggregate regions were formed after 10 hours.

4) The present examination suggested that the cellular structure of Ti-rich area, typically with the cell size of

~0.02 mm, was already formed by the welding. PWHT enhanced the heterogeneous precipitation in the Ti-enriched area absorbing the impurities in the surrounding Ti-depleted area. The purification of the Ti-depleted area resulted in the recovery of mechanical properties.
5) The suggested PWHT condition to effectively form the heterogeneous precipitates and purify the matrix is 1073 K and one hour, within the present test matrix.

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