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# The microstructure of laser welded Y doped V-4Cr-4Ti alloys after ion irradiation

H. Watanabe<sup>a,\*</sup>, A. Higashijima<sup>b</sup>, N. Yoshida<sup>a</sup>, T. Nagasaka<sup>c</sup>, T. Muroga<sup>c</sup>

<sup>a</sup> Research Institute for Applied Mechanics. Kvushu University, 6-1, Kasuga-kouen, Kasugasi, Fukuoka 816-8580, Japan

<sup>b</sup> Interdisciplinary Graduate School of Engineering Science, Kyushu University, 6-1, Kasuga-kouen, Kasugasi, Fukuoka 816-8580, Japan

<sup>c</sup> National Institute for Fusion Science, 322-6, Oroshi, Toki, Gifu 509-5292, Japan

## ABSTRACT

Laser welded V–4Cr–4Ti–0.15Y alloy, fabricated by National Institute for Fusion Science (NIFS), was used in this study. Copper ion irradiation was carried out with the tandem accelerator at Kyushu University. The TEM samples were sliced from the welded materials and irradiated at 873 K up to the dose of 12 dpa. The microstructure before the irradiation showed that relatively large precipitates, which were commonly observed in the alloy, disappeared in the center of the weld metal. After the ion irradiation, fine titanium oxides with {100} habit planes were detected. However, in all irradiation doses, the growth of titanium oxides was suppressed by Y addition.

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#### 1. Introduction

The welding procedure is one of the key technologies for use of V-4Cr-4Ti alloys as a large component [1]. But the embrittlement caused by interstitial impurities during welding is highly pronounced. To avoid the pick-up of impurities (e.g. oxygen and nitrogen) from the welding environment, electron beam (EB) and gas tungsten arc (GTA) welding [2,3] were conducted using vacuum chamber or glove box. Recently, laser welding technology for the alloys was developed by NIFS (National Institute for Fusion Science) by controlling the flow rate of high purity argon gas [4,5]. Because of flexible, in-field, automated and remote operation, and small weldment and heat affected zone (HAZ), laser welding is an attractive welding technology. However, quite little is knows as to the irradiation effect on the weldment. Our previous studies [6] on neutron irradiated V-4Cr-4Ti alloy (NIFS-HEAT2) revealed that tiny Ti(CON) precipitates were homogenously formed in the weld metal at 673 K and the formation was prominent in comparison with base metal. The effects of post-weld heat treatment (PWHT) on weld metal, effectively improving the CVN impact properties for unirradiated material and for material irradiated at lower temperatures, are not effective or have a very limited effect at higher irradiation temperatures where the growth of Ti(CON) precipitates were prominent. On the other hand, Y addition on V-4Cr-4Ti alloys is expected to reduce the Ti(CON) formation, because oxygen is scavenged by Y. The present paper summarized, therefore, the microstructural evolution of laser welded Y doped V-4Cr-4Ti alloy during ion irradiation.

Corresponding author.
E-mail address: watanabe@riam.kyushu-u.ac.jp (H. Watanabe).

#### 2. Experimental procedure

Welded joints used in this study were prepared from V–4Cr–4Ti–0.15Y alloy. Before the YAG laser welding (bead-on-plate welding) in a high purity argon atmosphere, the samples were annealed in a vacuum at 1273 K for 2 h. The detailed welding procedure was described elsewhere [3]. A 2.4 MeV copper ion irradiation was carried out with the tandem accelerator at Kyushu University. The TEM samples were sliced from welded materials and irradiated at 873 K up to the dose of 12 dpa. After the irradiation, the specimen was electro-polished by a back thinning method, and the area near the peak damage region (at about 700 nm) was observed by TEM. The damage rate and the implanted copper concentration in this region were  $1.7 \times 10^{-4}$  dpa/s and  $10^{-2}$  at.% (at 1 dpa), respectively.

### 3. Results and discussion

#### 3.1. Microstructure of unirradiated samples

Fig. 1 shows microstructural development of the weld metal with a distance from the bead center. In the figure, microstructure of the base metal is also shown. Ti enriched blocky precipitates, which were commonly observed in the base metal and composed of titanium, carbon, nitrogen and oxygen, disappeared in the weld metal (bead center). And relatively higher dislocation density was observed in the weld metal and the heat affected zone (HAZ).

As shown in Fig. 2(a), an increase in hardness occurred in the weld metal and also in the heat affected zone. namely, the hardness of weld metal was about 330 Hv, but the value suddenly dropped from 330 Hv to about 150 Hv at 4.5 mm from the bead center. Fig. 2(b) shows the measured dislocation and blocky



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Fig. 1. Microstructure of laser welded V-4Cr-4Ti-0.15Y alloy.



Fig. 2. Measured hardness, precipitate density and dislocation density near bead center.



Fig. 3. Microstructural evolution of laser welded V-4Cr-4Ti-0.15Y alloy and NIFS-HEAT2 at 873 K.

precipitates density near bead center. The hardening of the samples was explained by the disappearance of the blocky precipitates which resulting in an increase in the concentration of interstitial impurities (namely oxygen) and/or titanium.

## 3.2. Effects of Y addition on microstructure

Fig. 3 shows the microstructural evolution of base metal and weld metal of V-4Cr-4Ti-0.15Y alloy irradiated at 873 K. In the



Fig. 4. Measured size and density of Ti(CON) precipitates at each irradiation doses.

figure, microstructure of V-4Cr-4Ti (NIFS-HEAT2) is also shown for comparison. In the case of NIFS-HEAT2, fine titanium oxides with {100} habit planes were observed even at the dose of 0.75 dpa. The number density of Ti(CON) decreased with increasing dose and the growth of Ti(CON) precipitates was prominent at higher dose levels above 4 dpa. Estimated oxygen levels from the measured density and size of Ti(CON) precipitates were inserted in Fig. 3. In this estimation, Ti(CON) precipitates are assumed to be TiO (NaCl type crystal structure). For the case of NIFS-HEAT2, the estimated oxygen levels from the microstructure increased with dose and the value of the sample irradiated at 12 dpa is about 20 times higher than that of unirradiated sample. In V-4Cr-4Ti-0.15Y alloy, on the other hand, Ti(CON) precipitates were observed in all dose levels but smaller Ti(CON) precipitates were observed, in comparison with NIFS-HEAT2. The estimated oxygen levels of the base were almost half of NIFS-HEAT2. But, in the higher dose level above 7.5 dpa, the growth of Ti(CON) became prominent. The same oxygen pick-up from vacuum environment during ion irradiation is also reported in Ref. [7]. Therefore, in higher dose levels, oxygen pick-up from irradiation environment is essential, and



Fig. 6. Irradiation dose dependence of hardness at 873 K.

thus further studies are needed on Ti(CON) formation (oxidation kinetic) during irradiation (see Fig. 4).

## 3.3. Effects the PWHT on joint

As shown in the previous section, the formation of Ti(CON) precipitates is detected in the joint of V-4Cr-4Ti-0.15Y alloy. In the case of NIFS-HEAT2, it is known that the irradiation hardening of weld metal was effectively reduced by PWHT at 1073 K. The PWHT on NIFS-HEAT2 is very effective for relatively lower temperature irradiation (below 573 K). Because a highly segregated distribution of Ti(CON) precipitates were formed during the PWHT at 1073 K. Namely, after the annealing, microstructure of the weld metal was divided into two regimes, precipitates-segregation (PS) and precipitates-free (PF) areas. The formation of PS and PF areas is very important to reduce the formation of Ti(CON) precipitate during irradiation, because oxygen impurities are segregated in PS areas, and the PF areas were purified by PWHT. Fig. 5 shows the microstructure, of V-4Cr-4Ti-0.15Y joint after the annealing at the temperature range of 873–1073 K for 1 h. In the figure, Vicker's hardness of each samples were inserted in the figure.



Fig. 5. Annealing temperature dependence of V-4Cr-4Ti-0.15Y (weld metal, annealing time; 1 h). Upper and lower photos show the images taken by lower and higher magnification, respectively.

As shown in the figure, after the annealing at 1073 K for 1 h, PS and PF areas were formed and hardness of the weld metal was reduced to about 250. Fig. 6 shows the hardness changes after the irradiation at 873 K. It was shown that PWHT at 1073 K is contribute to reduce the hardness after the irradiation at all dose levels up to 12 dpa. Neutron irradiation studies using JOYO is in progress to confirm the effective of PWHT on Y doped V–4Cr–4Ti joints.

## 4. Summary

Copper ion irradiation was carried out on laser welded V–4Cr– 4Ti–0.15Y alloy. The main results are summarized as follows.

- (1) The microstructure before irradiation showed that relatively large precipitates disappeared in the center of weld metal.
- (2) After the ion irradiation at 873 K, fine titanium oxides with {100} habit planes were detected even at the dose of 0.75 dpa. This means that the behaviors of oxygen atoms, which dissolved from the large precipitates during the laser welding, is essential to the microstructural evolution of V– 4Cr–4Ti–0.15Y alloys.
- (3) At 873 K irradiation, effects of oxygen from irradiation environment are essential, especially at higher dose levels. Further studies are needed on oxygen pick-up and oxidation kinetic during irradiation.

(4) The understanding the effects of PWHT on weld metal is important to reduce the radiation hardening of the materials.

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