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The microstructure of laser welded V–4Cr–4Ti alloy after neutron irradiation

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Abstract

Effects of neutron irradiation on YAG laser welded V–4Cr–4Ti alloy were studied in JMTR (Japanese Materials Testing Reactor). The samples with and without post-weld heat treatment (PWHT) at 1073 K were irradiated in the temperature range of 673–873 K up to the dose of 0.45 dpa. After the irradiation, the microstructure and Vickers hardness of the welded samples were compared of the base metal, which were simultaneously irradiated at the same irradiation cycle.

1. Introduction

It is recognized that welding procedure is one of the key technologies for use of V–4Cr–4Ti alloys in a large component [1]. However the susceptibility of these alloys to 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 a vacuum chamber or a 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]. Because of the flexible, in-field, automated and remote operation, and small weldment and heat affected zone (HAZ), laser welding is an attractive welding technology.

Our recent ion irradiation on these materials [5] revealed that the defect cluster density (mainly interstitial type dislocation loops) of weld metal is almost comparable to that of base metal after ion irradiation at 573 K. On the other hand, after irradiation at 873 K, fine titanium oxides with {1 0 0} habit planes were detected even at the dose of 0.75 dpa. This means that the behavior of oxygen atoms, which dissolve from the large precipitates during laser welding, strongly affects microstructural evolution of welded V–4Cr–4Ti alloys during irradiation at 873 K.

However, little is known regarding the effects of neutron irradiation on the weldment. Nagasaka et al. [6] revealed that weld metal showed larger

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irradiation hardening than that of the base metal after neutron irradiation at 563 K. The hardening of the weld metal was effectively reduced by post-weld heat treatments (PWHT) at 873 K and 1223 K. The irradiation hardening at 563 K was mainly controlled by a very high density of dislocation loops, but in higher irradiation temperature regimes, formation of radiation-induced Ti(CON) precipitates becomes dominant. The present paper summarizes the recent progress on the microstructural evolution of laser welded V–4Cr–4Ti alloy during neutron irradiation at higher irradiation temperature regimes.

2. Experimental procedure

Welded joints used in this study were prepared from a high purity V–4Cr–4Ti alloy, which was designated as NIFS-HEAT-2 [1,7]. Before the YAG laser welding (bead-on-plate welding) in a high purity argon atmosphere, the samples were annealed in vacuum at 1273 K for 2 h. The detailed welding procedure was described elsewhere [3]. Oxygen concentrations of the sample before and after welding were 139 and 158 wt ppm, respectively. The PWHT was carried out in a vacuum of about 1×10^{-5} Pa at 1023 K for 1 or 100 h. Before the annealing, the samples were sandwiched between tantalum sheets and they were wrapped in zirconium foil. The samples were encapsulated in quartz glass to prevent the contamination of impurities. Fission neutron irradiation was carried out in JMTR under improved temperature control condition at 673, 723 and 873 K in the same irradiation cycle (namely, JMTR 03 M-69U). The total neutron dose of irradiation was $5.18 \times 10^{24}/m^2$ (>1.0 MeV), which corresponds to 0.45 dpa.

3. Results

3.1. Temperature dependence of microstructure

The microstructure of laser welded NIFS-HEAT-2 was strongly dependent on irradiation temperature. The upper part of Fig. 1 shows the TEM images of base metal (a), and weld metal (b), after irradiation at 673 K. The corresponding images of base metal and weld metal which were post-weld heat treated at 1073 K for 100 h are also shown. The figure shows the radiation enhanced formation of Ti(CON) precipitates with {1 0 0} habit planes and dislocations. Ti(CON) precipitates were homogenously formed in the weld metal. The measured number density of precipitates in the base metal and weld metal were 1.4×10^{21} and 4.3×10^{21} (m^{-3}), respectively. The average precipitate size in the base metal and weld metal was 44 and 18 (nm), respectively. From our previous results [8], a highly

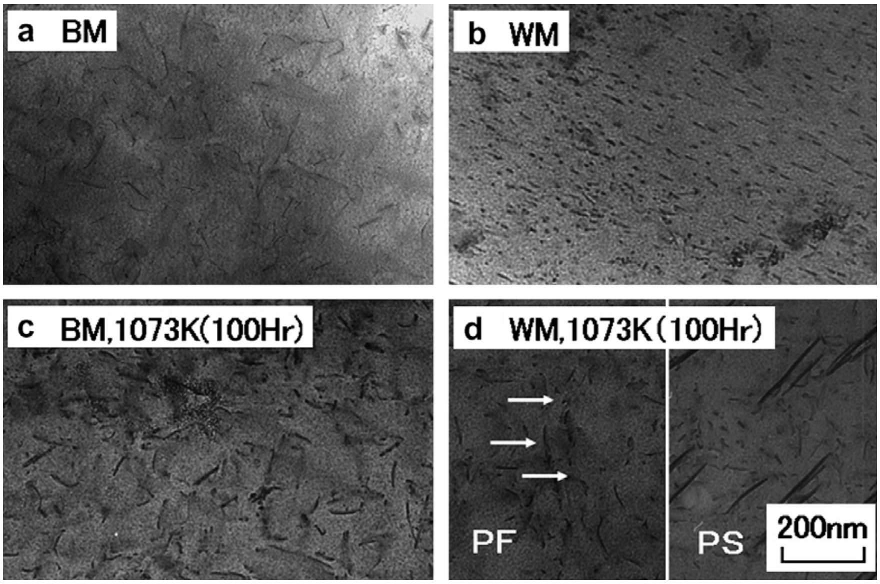

Fig. 1. Microstructure of the samples irradiated at 673 K: (a) base metal, (b) weld metal, (c) base metal with PWHT (1073 K, 100 h) and (d) weld metal with PWHT (1073 K, 100 h).

segregated distribution of Ti(CON) precipitates were formed during the PWHT at 1073 K. Namely, after the annealing, the microstructure of the weld metal was divided into two regions, precipitate–segregation (PS) and precipitate-free (PF) areas. As shown in Fig. 1(d), after the irradiation at 673 K, relatively larger Ti(CON) precipitates were observed in PS areas and small precipitates of about 40 (nm) (shown by arrows in the figure) were also observed in PF areas of the weld metal.

Fig. 2 shows the microstructure of weld metal irradiated at 873 K. At this temperature, PS and PF areas were also observed in weld metal without the PWHT at 1073 K. Fig. 3 shows the microstructure of base metal (a), weld metal (b); base metal and weld metal which were given the PWHT of 1073 K (100 h) are shown in (c) and (d). Relatively large Ti(CON) precipitates of about 200 (nm) and tiny Ti(CON) precipitates of about 10 (nm) were observed together in all samples. Size distributions of precipitates for the sample of base metal and weld metal are shown in Fig. 4(a) and (b), respectively. In the figure, measured values of the samples irradiated at 673 K are also shown. The precipitate number density and average size of precipitates for specimens irradiated at 673 K and 873 K are summarized in Table 1.

3.2. Hardness changes and microstructure after the irradiation at 723 K

Fig. 5 shows the microstructure after irradiation at 723 K. In the left corner of each photo, void contrast images ($s \gg 0$) taken by higher magnification are inserted. After the irradiation at 723 K, PS and PF areas were also formed in weld metal without PWHT. But higher magnification of void contrast images revealed that precipitates also existed in PF area in weld metal with and without PWHT (shown by arrows in Fig. 5(b) and (c)).

Fig. 6 shows the hardness distribution around the weld bead centre before and after the irradiation at 723 K. From the microstructural observations, the width of the weld metal was 1 mm as indicated in the figure. At 723 K, Vickers hardness of base metal was almost comparable to that of weld metal in the figure, hardness changes due to the irradiation at 563 K, are also shown for comparison [8]. At 563 K, irradiation hardening of the weld metal was relatively larger than that of the base metal, but at high irradiation temperatures, irradiation hardening of the base metal became prominent. The PWHT at 1073 K was very effective before and even after the irradiation at 563 K [8]. These results mean that the PWHT improved the CVN properties for both the unirradiated material and

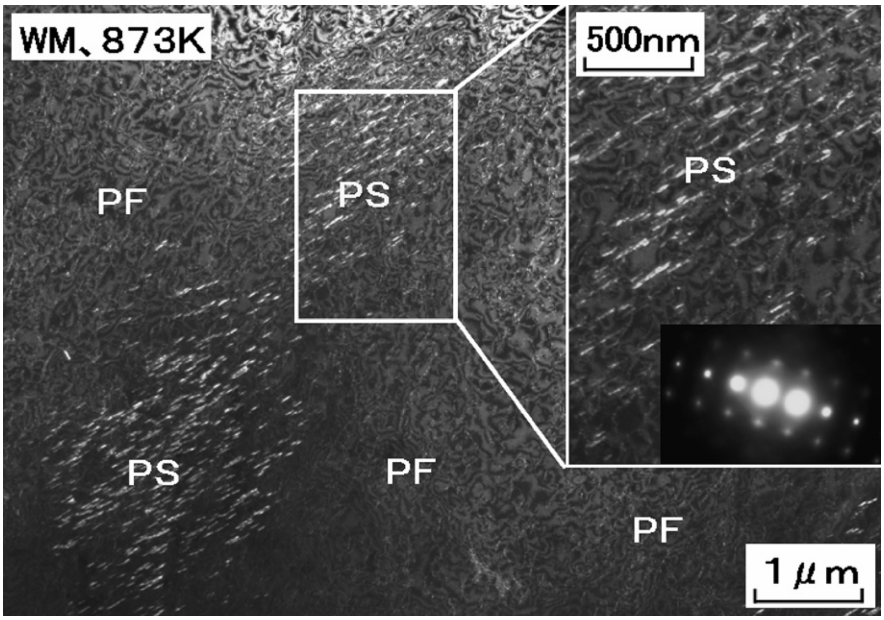

Fig. 2. Dark field images of weld metal irradiated at 873 K. At this temperature, PS and PF areas were also observed in weld metal without the PWHT.

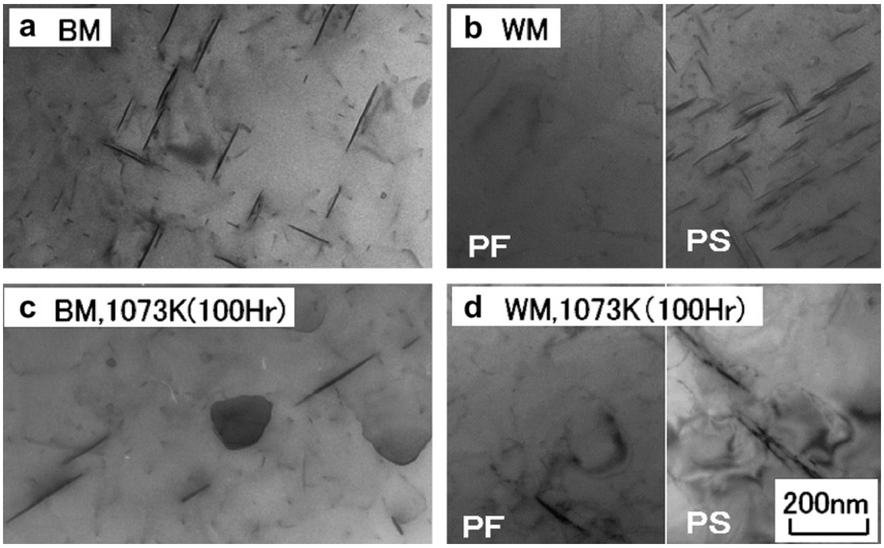

Fig. 3. Microstructure of the samples irradiated at 873 K: (a) base metal, (b) weld metal, (c) base metal with PWHT (1073 K, 100 h), (d) weld metal with PWHT (1073 K, 100 h).

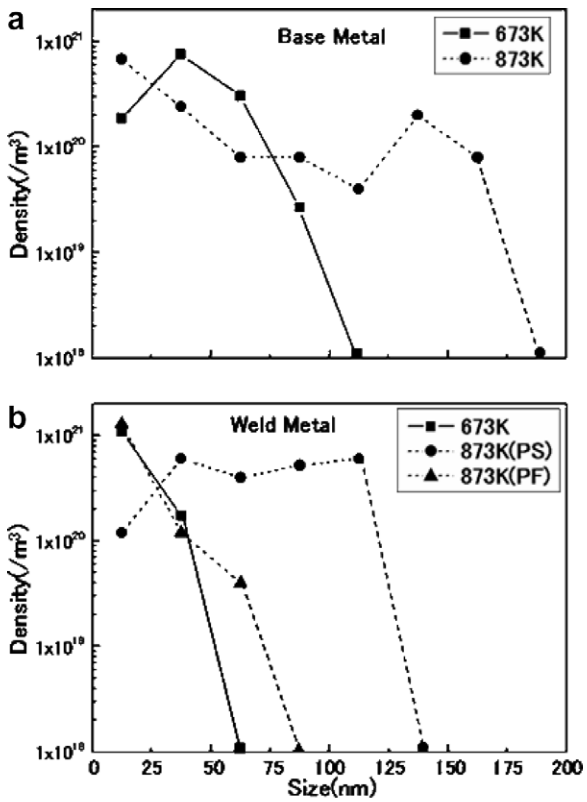

Fig. 4. Measured precipitate size distributions of the samples irradiated at 673 K and 873 K: (a) base metal and (b) weld metal.

for the materials irradiated at 563 K, as shown in the related work [8].

4. Discussion

In NIFS-HEAT-2, it is known that blocky (larger) Ti-rich and small Ti–C–O precipitates existed in the base metal before laser welding. Theses precipitates were dissolved in the weld metal during the welding procedure, resulting in an increase in the concentration of interstitial impurity (mainly oxygen) and/or titanium. The increase in hardness in the as-welded condition could be explained by the interstitial impurity in solid solution in the matrix. The PWHT at 1073 K results in an increase in the absorbed energy during impact testing of welded samples by forming PS and PF areas in the weld metal [8]. From our previous studies using the identical samples [5,6], at lower irradiation temperatures, radiation induced-hardening of welded samples can be quantitatively explained by the number density and size of radiation induced defects (dislocation loops and/or precipitate) using Orowan's equations. For the case of ion irradiation at 573 K, for example, the number density of defects in weld metal and base metal were almost the same from 0.75 to 7.5 dpa, but the measured hardness in the weld metal was about 10% higher than that of base metal. Besides dislocation loops, very small Ti(CON) precipitates of about 1–2 nm were detected in the weld metal by high resolution electron microscopy (HRTEM) [5]. This suggests that the additional radiation hardening in the weld metal

Table 1
Measured precipitate number density and averaged size of precipitates of specimens irradiated at 673 K and 873 K

		Base metal	Weld metal	
673 K irradiation				
Without PWHT	Size (nm)	43.5	17.7	
	Density ($/m^3$)	1.4×10^{21}	4.3×10^{21}	
PWHT (1073 K, 100 h)	Size (nm)	31.0	PS	PF
			41.0	35.4
	Density ($/m^3$)	3.1×10^{21}	4.6×10^{21}	1.9×10^{21}
873 K irradiation				
Without PWHT	Size (nm)	51.0		
			71.8	12.8
	Density ($/m^3$)	1.4×10^{21}	2.3×10^{21}	1.4×10^{21}
PWHT (1073 K, 100 h)	Size (nm)	38.5		
			144.0	33.7
	Density ($/m^3$)	9.6×10^{20}	8.9×10^{19}	4.1×10^{20}

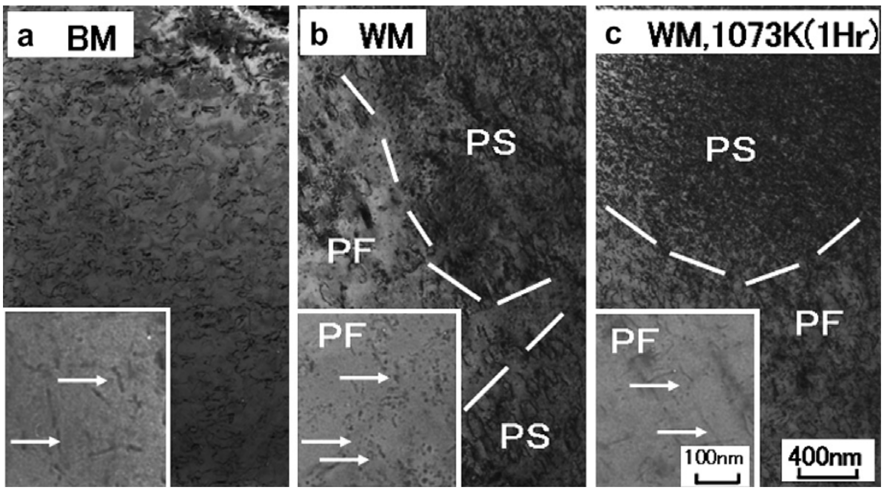

Fig. 5. Dislocation contrast images of the samples irradiated at 723 K: (a) base metal, (b) weld metal, (c) weld metal with PWHT (1073 K, 1 h). In the left corner of each photo, void contrast images ($s \gg 0$) taken by higher magnification are inserted (precipitates are shown by arrows).

is presumably due to the enhanced formation of small Ti(CON) precipitates.

With increasing irradiation temperature, growth of Ti(CON) precipitates became prominent and the precipitate was easily identified by their habit planes. As shown in Fig. 1, for the 673 K irradiation, Ti(CON) precipitates were homogenously formed in the weld metal and the number density of Ti(CON) precipitates was about three times higher than that of the base metal. At temperature above 723 K, Ti(CON) precipitates were not formed uniformly. Namely, precipitate–segregation (PS) and precipitate-free (PF) areas, which were commonly observed after the PWHT at 1073 K, also appeared in the weld metal. At 873 K, large Ti(CON) precipitates were commonly observed in the PF areas in the weld metal. It is important to note that the absorbed energy of the welded sample increases significantly, when the microstructure is divided into PS and PF areas. But once the plate-like precipitates with typical {1 0 0} orientation are formed, the absorbed energy of welded sample drops drastically. Therefore, the effects of PHWT on weld metal, which are useful for unirradiated and lower temperature irradiations, are not effective or very limited at higher irradiation

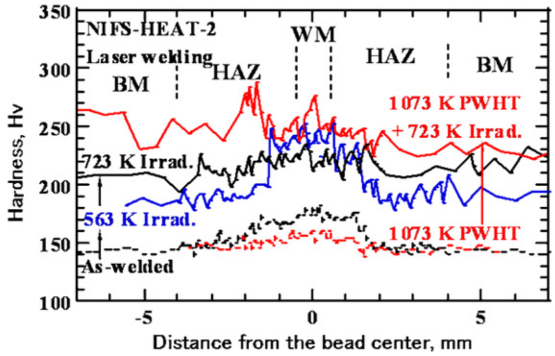

Fig. 6. Hardness distribution around the weld bead. The vertical dashed lines indicate regions of weld metal (WM), heat affected zone (HAS) and base metal (BM).

temperatures where plate like Ti(CON) precipitates are formed.

5. Conclusions

The microstructural developments and changes in Vickers hardness of laser-produced weldment due to neutron irradiation at 673–873 K were investigated with and without a PHWT at 1073 K. The main results are summarized as follows:

(1) At 673 K, tiny Ti(CON) precipitates were homogenously formed in the weld metal and the formation was prominent in comparison with base metal. Above 723 K, the microstructure of the weld metal was divided into two regions precipitate–segregation (PS) and precipitate-free (PF) area.
(2) After the irradiation at 723 K, in the base metal, radiation hardening due to the formation of dislocations and Ti(CON) precipitates became prominent. The hardness of the base metal was almost the same level as the weld metal.
(3) The effects of PHWT 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 plate like Ti(CON) precipitates were formed.

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