Experimental assessment of temperature distribution in heat affected zone (HAZ) in dissimilar joint between 8Cr-2W steel and SUS316L fabricated by 4 kW fiber laser welding

Sho KANO*, Akira OBA**, Huilong YANG*, Yoshitaka MATSUKAWA*, Yuhki SATOH*, Hisashi SERIZAWA***, Hideo SAKASEGAWA****, Hiroyasu TANIGAWA**** and Hiroaki ABE*

*Institute for Materials Research, Tohoku University,
2-1-1 Katahira, Sendai 980-8577, Japan
E-mail: kano.sho@imr.tohoku.ac.jp

**Graduate School of Engineering, Tohoku University,
6-6-01-2Aobayama, Sendai 980-8579, Japan

***Moriba Joining and Welding Research Institute, Osaka University,
11-1 Mihogaoka, Ibaraki, Osaka 567-0047, Japan

****Fusion Research and Development Directorate, Japan Atomic Energy Agency,
2-166 Omotedate, Obuchi, Rokkasho, Aomori 039-3212, Japan

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Abstract

High-Cr steel (Fe-8Cr-2W-0.4Mn-0.2V-0.06Ta, so-called F82H steel) is one of the candidate structure materials for fusion blanket system, and its dissimilar welding technology to stainless steel (SUS316L) is an inevitable issue. Fiber laser welding is a promising technique to joint F82H and SUS materials, because of its non-mandatory vacuum environment during welding, and the reduced distortion and residual stress. Purpose of the present study is to identify the microstructure characteristics of dissimilar joint between F82H and SUS316L fabricated by fiber laser welding. $M_{23}C_6$ -type precipitates were evaluated using an electron back-scatter diffraction device equipped with a scanning electron microscope, an electron probe micro analyzer, and a transmission electron microscope. The microstructure of as-welded specimens was composed of heat affected zone (coarse grain (CGHAZ) and fine grain (FGHAZ)) in F82H side, and weld metal (WM). Distributions of size, density and chemical composition of precipitates in HAZ were examined as a function of the distance from WM/HAZ interface. Besides, isochronal annealing experiments on F82H performed to obtain relationship between annealing temperature and Cr (or C) concentration in $M_{23}C_6$ precipitates. Cr-rich $M_{23}C_6$ comparing with original F82H (tempered F82H) one was observed at temperature ranging from 1103 to 1173 K. By comparison of their microstructure with that of HAZ, temperature distribution in HAZ upon welding was evaluated. It is suggested that the HAZ/F82H interface and the FGHAZ/CGHAZ boundary were heated up to 1150 K and 1400 K, respectively. Further applications of this method to clarify the temperature history and its distribution in HAZ, as well as increase in diagnostic accuracy, are greatly expected.

Key words : Fiber laser welding, Dissimilar joint, F82H, SUS316L, $M_{23}C_6$, Temperature gradient, HAZ

1. Introduction

As one of the reduced activation ferrite/martensitic steels (F82H) has similar to commonly 8-9 Cr type heat-resistant steel, and it is regarded as the promising candidate material for the Japanese ITER test blanket module (TBM) system (Hashimoto et al. (1998)). TBM system is composed of complex and many integrated functions, materials and interfaces (Enoeda et al. (2012)) such as first wall, side and back plates, coolant pipes and so on. Dissimilar welding of F82H is

inevitable for the fabrication of the blanket system. One of the examples is the water-cooling pipes that are welded to ITER water cooling system made by SUS316L (SUS).

To date, investigations on welded F82H have been carried out. Techniques such as tungsten inert gas (TIG) welding (Tanigawa et al. (2003)), friction stir welding (FSW) (Hoelzer et al. ((2013))), electron beam (EB) welding (Hara et al. (2009)) and fiber laser welding (Serizawa et al. (2013)) have been applied for the fabrication of dissimilar weld joint between F82H and SUS. The fiber laser welding technique is currently regarded as one of the most desirable one because of its high speed and deep penetration as well as high efficiency, high power and high beam quality, enabling to achieve ultra-high peak power density, such as the order of MW/mm (Kawahito et al. (2007)). Serizawa et al. (2013) reported that the fiber laser welding is a suitable way to joint F82H and SUS materials, because the vacuum environment is not mandatory during welding, and the distortion and residual stress by welding is reduced. However, most of the research has been focused on welding process optimization, mechanical properties evaluation, the effect of post-weld heat treatment and irradiation characteristics. Less attention has been paid on the welding between F82H and SUS in terms of microstructure evaluation which affects mechanical strength significantly.

It is commonly believed that a key factor that influences its creep and irradiation integrity is the microstructure of HAZ in F82H welded component (Jia and Dai (2005)), particularly the precipitates, such as $M_{23}C_6$ and MX (Abe et al. (1992), Sawada et al. (2003)). For example, the particles embedded in matrix are able to prevent the grain coarsening effectively; and, the finely-dispersed precipitates significantly contribute to the mechanical properties (Armaki et al. (2013)). General understanding of $M_{23}C_6$ (M=Cr, W and Fe) and MX (M=Ta, V; X=C, N) is that they are precipitated during tempering whose temperature is ranged roughly from 1013 K to 1053 K. Therefore, the precipitates in F82H would undergo distinct change caused by the high heat input during the welding. The microstructure changes in HAZ including phase transformation and precipitation instability are closely related to the temperature and its history induced by the high energy laser source followed by the prompt cooling. However, it is extremely difficult to accurately measure the local temperature because of the steep temperature gradient in narrow width of HAZ, generally at the scale of hundreds of μm .

Thus, the purpose of present study is to investigate the microstructure, such as chemical composition precipitate, in HAZ in the dissimilar weld joint between F82H and SUS fabricated by a fiber laser. Further, it is our particular interest to assess experimentally the temperature gradient profile in HAZ upon welding by comparing the precipitation behaviors in annealed F82H specimens. To our knowledge, this is the first-time approach that allows experimental evaluation of the temperature history and distribution upon welding.

2. Experimental procedure

F82H and SUS plates whose size was 100 mm in length, 50 mm in width, and 4 mm in thickness were used as experimental materials. The chemical composition of the F82H is 0.1 C, 0.1 Si, 0.44 Mn, 7.81 Cr, 1.84 W, 0.06 Ta, 0.2 V, and the balance Fe, in contrast, the chemical composition of SUS is 0.02 C, 0.73 Si, 0.99 Mn, 12.13 Ni, 17.25 Cr, 2.11 Mn, the balance Fe (all in wt.%). F82H was normalized at 1313 K for 0.5 h and tempered at 1013 K for 1.5 h. This tempering history is slightly different from the original one (Okubo et al. (2011)); slightly lower temperature (-10 K) and longer isothermal holding time (+0.5 hr).

^{#1, 2)} A 4 kW fiber laser with the wavelength of 1070 nm and the beam diameter of 200 μm was used as a probe beam for laser welding. And the gas flow and focal length of laser beam were 40 L/min and 0 mm, respectively. Also the

welding speed was 4 m/min and the laser beam position was right on the F82H/SUS contact surface. The detailed parameters on the fiber laser welding are listed in the Serizawa et al. (2013).

The precipitation behaviors in F82H under the thermal annealing condition were investigated in F82H exposed to annealing at various temperatures 1073, 1103, 1123, 1153, 1173, 1193, 1223, 1253 and 1323 K for 40 min in vacuum at the level as low as 10^{-3} Pa followed by cooling in air. The heating and cooling rates were 3 and 2 K/s, respectively.

Microstructure characterization was performed by field emission scanning electron microscope (FE-SEM), electron probe micro analyzer (EPMA), electron back-scatter diffraction (EBSD) and transmission electron microscope (TEM). For both EPMA and EBSD, 20 kV as accelerating voltage of electrons and $^{1,3)} 1 \mu\text{m}$ as beam spot diameter and $0.06 \mu\text{m}$ as scanning step were commonly chosen. The electron-transparent TEM foils, achieved by electrochemical polishing in 8% perchloric acid and acetic acid at room temperature, were taken from the center of HAZ.

3. Results and discussion

By SEM observations, it was clearly seen that the as-welded specimen was classified into four distinct parts; namely, base metal of F82H (F82H), heat affected zone (HAZ) in F82H, weld metal (WM), and base metal of SUS (SUS). The average HAZ width was approximately $200 \mu\text{m}$. The geometric information about EPMA analyses locations from 1 to 7 were 266, 205, 156, 110, 48, 15 and $-84 \mu\text{m}$ away from WM/HAZ interface, respectively.

Figure. 1 shows the results of EBSD microstructural analysis of HAZ and its vicinity. Microstructural features such as grain size and shape and internal strain are as follows. The grain size and internal strain nearby the HAZ/F82H interface was similar to the F82H matrix. In the HAZ region, relatively fine grains and coarse grains were observed in 0-145 and 145-180 μm away from F82H/HAZ interface, respectively. Kato et al. (2009) pointed out that the HAZ in F82H formed by EB welding could be categorized into three regions; coarse grain HAZ (CGHAZ), fine grain HAZ (FGHAZ) and over tempered HAZ (THAZ). According to the reference (P. Mayer (2007)), the heating history of HAZ is described as follows: *CGHAZ region adjacent to the fusion line experienced temperatures well above the A_{c4} temperature, and delta ferrite (δ)*

Figure 1 (a) Optical microscopy image and (b) inverse pole figure (IPF) map of the area peripheral to HAZ region of as-welded specimen. (c) The grain reconstruction image of selected area in (b), showing the distribution of fine γ grain. The grain boundary (black line) defined misorientation angle above 2 degrees.

grains nucleated and grew at highest peak temperature ($T > 1523$ K). Also, the small-size austenitic grains were formed by lower temperature annealing, which is presumed to be just above A_{c3} in FGHAZ. The growth of martensite-block grains occurred in THAZ region, because of over-recovery caused by the high temperature annealing comparing to the tempering regime of fabrication process of the steel; i.e. 1023 K for 60 min. ^{#1,1)} Therefore, the CGHAZ with the average grain size of 2 - 4 μm nearby the WM region and FGHAZ with the average grain size of ~ 0.5 μm situated approximately 30 μm away from WM/HAZ interface could be clearly distinguished, although the THAZ was not obvious as shown in the Fig. 1(a).

In addition to the above-mentioned over-all characteristics of HAZ microstructure, fine and condensed grains with the diameter of approximately 0.7 μm were observed typically in 172-192 μm away from WM/HAZ interface. They were anticipated the recovered martensitic-block grains formed by the annealing during welding at the temperature range where $\alpha+\gamma$ phase was stable. The other characteristic is the fine grains, formed within lath or its interface, which were typically observed in 125-160 μm away from WM/HAZ interface. The fine grains are emphatically shown in red shown in Fig. 1(b), which is the grain reconstruction map from the dash-dotted area in Fig. 1(a). They were presumably γ -phase grains which can be formed during welding when the temperature was spontaneously higher than the A_{c1} (Tanigawa et al. (2003)). The nucleation of such fine grains seemed preferential nearby the grain boundary, which can be clearly found from Fig. 1(b). This tendency is generally consistent with the previous report where Shirane et al. (2009) revealed that α to γ reverse transformation in a 9Cr-2W type heat-resistance (Gr92) steel occurred at prior austenitic grain boundary. A new insight derived in this work is that the reverse transformation occurred preferentially at martensitic-lath grain boundary in F82H.

Figure 2 shows the Cr and C mapping results of F82H and derived from various locations 2-6 in HAZ. The arrayed and particle shaped segregations of Cr and C in F82H specimen were clearly observed. According to the previous studies (Sakasegawa (2011)), $M_{23}C_6$ in the original F82H steel were formed on grain boundary by EPMA. Thus it is conceivable that the segregated particles are $M_{23}C_6$.

In the locations 2-6 in HAZ, the Cr segregation was clearly seen in F82H (location 2). Intensity of Cr gradually decreased when the position shifted to weld metal (WM), and barely detected at location 6. On the other hand, it is

Figure 2 Cr and C mapping results of F82H matrix and derived from various locations 2-6 in HAZ of as-welded specimen.

Figure 3 Average Cr and C concentrations in precipitates as a function of the distance from WM/HAZ interface. Error bars indicate the standard deviation of measurement from 30 precipitates for each location.

noteworthy that the C segregation was only detected in the area nearby by the F82H/HAZ interface (locations 2 and 3). With respect to the observations, therefore, it is presumed that the metallic carbide $M_{23}C_6$ was present in the locations 1-3, Cr-rich precipitates in the locations 4-5, and absent of precipitates in the location 6.

In order to evaluate the precipitation behavior of $M_{23}C_6$ in HAZ, the distributions of Cr and C concentrations measured from corresponding mapping results are shown in Figure 3. 30 precipitates for each location were measured, from which the average concentration of precipitate and standard deviation were assumed. Note that the averaged concentration of Cr and C from whole of the mapping area was 12.02 and 0.40 wt.% respectively. On the other hand, the Cr and C concentrations of precipitates in F82H were analyzed as 14.52 and 0.76 wt.%, respectively. Segregation of Cr at the precipitates and depletion of C were observed. TEM/EDS analysis (Lu et al. (2009)), the chemical composition of Cr and C of $M_{23}C_6$ in F82H was 61 and 13 wt.%, respectively. This difference is probably attributable to the experimental and/or instrumental error. Characteristic X-rays detected in EPMA contains signals from a certain volume which is much larger than the probe size due to the scattering the electron beam inside specimen (Kanaya et al. (1972)). The probe size and sampling step of EPMA were ≈ 100 nm and 60 nm, respectively, and the average particle size of $M_{23}C_6$ in F82H was approximately 100 nm (Jia and Dai (2004)). Therefore, one should expect the measured composition contains much information from both precipitates and matrix. This attributes to lower the concentration of Cr and C in the case of F82H. Furthermore, the effect of precipitate size on average concentration estimated from EPMA was evaluated in the present study. Results showed that the C and Cr concentration of precipitate increased with increasing of precipitate size; the precipitate size and area density decreased with increasing temperature. It was indicated that the size of precipitate shrunk with increasing temperature. In addition, in terms of the limitation of this method, the C and Cr concentration of size of precipitate less than ≈ 180 nm was general argument with the averaged concentration of Cr and C from whole of the mapping area; i.e. Cr and C concentration were 12.1 and 0.10 wt.%, respectively. Thus, the limitation size of precipitate in the present study's method is approximately 180 nm, even though the value of limitation size is depending on the image resolution. In conclusion, the values of concentration are incorrect quantitatively, nevertheless their relative variation is meaningful. Moreover, EPMA allows us to measure tens or hundreds of precipitates at once, which enhances statistical accuracy.

Overall, Cr and C concentration of in precipitates were 14.52 and 0.76 wt.% in F82H side, respectively. The

Figure 4 Cr and C mapping results of thermal equilibrium annealed specimen by EPMA. Annealed condition of (a), (b), (c) and (d) are 1073, 1153, 1173, and 1253 K for 40 min followed by air cooling, respectively.

Figure 5 Concentration of Cr and C in precipitates as a function of annealing temperature. Error bars indicate standard deviation of measurement 30 particles for each temperature.

concentration was maintained in HAZ closer to F82H, then it dropped until it reached to 12.2 and 0.08 wt.%, respectively. Obviously, it is found that the chemical composition of precipitates or the segregation Cr and C in HAZ is closely related to the distance from WM/HAZ interface.

From TEM observation, a particle with the diameter more than 250 nm was observed in the grain interior, which is 102 μm away from WM/HAZ interface (location 4). Based on the selected area diffraction analysis, an FCC-based structure precipitate with the lattice parameter of 1.05 nm was identified. This value is quite close to the literature where 1.06 nm was reported for M_{23}C_6 type particle in F82H (Jia and Dai (2004)). Besides, Chemical composition of Cr and C of them evaluated from EPMA were 14.5 and 0.82 wt.%, respectively. Thus, it is convinced that the M_{23}C_6 type precipitate is identified in the location 4 of HAZ. As anticipated by Fig. 3, the concentration of C the M_{23}C_6 precipitate is lower than that is raw F82H (Lu et al. (2009)) indicating the formation of non-stoichiometric $\text{M}_{23}\text{C}_{6-x}$.

It has been well known that the microstructure formed during a welding process is different from that attained from the ideal thermal annealing condition, mainly because of the strong temperature gradient from the heat source to matrix side caused by spontaneously high heat input of welding. Where, the microstructure of HAZ is formed by high temperature, non-equilibrium heat treatment and rapid-quenching due to welding depending on the location away from heat source. In present study, the effect of achieving temperature distribution during welding focused because the microstructure formation mechanism of HAZ is strongly dependent on the temperature (Tanigawa et al. (2003), Kato et al. (2009)). Therefore, the temperature distribution in laser welding is investigated using annealed specimens in order to better interpret the precipitate stability.

Cr and C mappings of the specimens after annealing at 1073, 1153, 1173, and 1253 K are shown in Figure 4. Decrease in Cr segregation with increase in annealing temperature was observed. Cr was completely dissolved in matrix by the annealing at temperature above 1173 K, which is consistent with the Ac_3 temperature (1183-1213 K) (Sawai et al. (2002), Zheng et al. (2011)). Similar to Cr concentration, C segregation also decreased with an increase in annealing temperature. Therefore, dissolution of M_{23}C_6 in F82H was confirmed at temperature of 1173 K and above.

Similarly, Cr and C concentrations in precipitates were measured by EPMA, and subsequently plotted are plotted as the function of annealing temperature as shown in Figure 5. The Cr concentration gradually decreased with increasing temperature when the annealing temperature was equal to or lower than 1193 K, subsequently it was stabilized at roughly 12.2 wt.% when the annealing temperature was higher than 1223 K. On the other hand, C concentration had a similar tendency: it decreased with increasing annealing temperature and it remained stable above 1223 K.

The decrease in C concentration above 1073 K would be responsible to reverse transformation. Because the solid solution limit of C in α and γ -phase are < 0.1 and 9 at.%, respectively (Masalski (1990)). The parabolic reduction of C concentration at the temperature range from 1073-1173 K indicates that the $\alpha \rightarrow \alpha + \gamma$ reaction underwent gradually with increasing annealing temperature accompanying with continuous elution of C from precipitates to matrix. In other words, the decomposition of M_{23}C_6 was controlled by the volume fraction of γ phase. However the mechanism of decomposition of precipitates at this temperature range is unclear yet, the diffusion rate of C in the temperature range is extremely fast, i.e. $4.5\text{-}5.5 \times 10^{-8} \text{ m}^2/\text{s}$ (Load and Beshers (1966)). Thus the TEM observations on the reverse transformation behavior especially at the periphery of M_{23}C_6 , as well as size distribution and number density would be necessary to elucidate diffusion process of C from precipitate, and to describe the decomposition mechanism of M_{23}C_6 .

The mathematical relationship between the temperature and Cr and C concentration is capable to be achieved based on Fig. 5 from the four dimensions logistic curve fitting formula:

$$\text{CONC} = \frac{d+(a-d)}{1+(T/c)^b} \quad (1)$$

where CONC is the concentration of Cr and C in wt.%; T is the temperature in Kelvin; a , b , c and d are constants. This formula is commonly used for curve-fitting analysis, especially, it is reasonable to explain the empirical formula of S-curve trend. The empirical fitting equations for Cr and C concentration were established as $\frac{91.7+(15.8-91.7)}{1+(T/1173.6)^{12.1}}$ and $\frac{0.06+(1.0-0.06)}{1+(T/1168.6)^{93.9}}$, respectively. Note that the equations may not express the strict metallurgical reactions.

Variation of the annealing temperatures during the real-time welding associated with the distance from WM/HAZ interface was evaluated. From Fig. 5 and Equation (1), the empirical thermal equilibrium temperatures in various locations in HAZ were established on the location matrix and derived from various locations 2-6 in as-welded specimen. The equations above were employed in this discussion because of the ease of the following analysis accepting a certain error. The data of the location 5 (47.6 μm from WM/HAZ interface) and location 6 (15.4 μm) are absent, because no precipitate was detected there. Figure 6 shows the temperature evaluated from Eq. (1) for the each locations (1-4 and 7). Here, the melting point of iron, i.e.: 1811 K, was assumed for the temperature at WM/HAZ interface. Characteristics of the microstructure from Figs. 2 and 4 is also shown. Fig. 6 indicates the temperature variation in HAZ estimated by precipitate composition. The temperature of HAZ/F82H interface was estimated as 1150 K, and that of FGHAZ/CGHAZ boundary was 1400 K.

To our knowledge, this is the approach for the first time that allows experimental evaluation of the temperature history and distribution upon welding. This evaluation, unfortunately, gives relatively higher temperature at HAZ/F82H interface than Ac_1 that is generally the temperature of γ -phase formation. The error was about 30-40 K. This is presumably caused by the duration of isothermal annealing, i.e. 40 min in present study. The longer duration proceeds $\alpha \rightarrow \gamma$ reaction, in other words, dissolution of Cr and C into matrix decomposing M_{23}C_6 precipitates. The other factor to be concerned is the effect of extremely large energy input during laser welding, instantaneously, the instability of precipitate under the non-equilibrium condition was not investigated in present study, so that, those are to be investigated in the future study. Therefore, the approach to experimentally estimate the temperature history during fiber laser welding was proposed based on the microstructure evaluation, especially the chemical composition change of M_{23}C_6 particle. If we are made the difference in temperature between attained result and true one, it is concluded that this mechanism cause by the presence of distinct precipitation behavior between non-equilibrium and equilibrium annealing. And more, the findings in the present study are in particularly important when considering the instability of precipitate under the non-equilibrium thermal condition, although the necessary improvement is still needed to further confirm the accuracy of this assessment approach.

4. Conclusions

To clarify the microstructure in F82H/SUS316L joint fabricated by fiber laser welding, the precipitation of M_{23}C_6 and phase stability of matrix was investigated using EBSD, EPMA and TEM. Correlation of microstructure between as-welded and thermally aged

Figure 6 Dependence of temperature under fiber laser welding on distance from WM/HAZ interface calculated by Cr/C concentration in M_{23}C_6 precipitate.

F82H successfully deduce temperature history and distribution in HAZ.

The dissimilar joint can be divided into four parts in terms of microstructure, namely base metal of F82H, HAZ in F82H, WM and SUS. HAZ can be categorized into FGHAZ and CGHAZ, although THAZ was not identified. $M_{23}C_6$ were observed in the area near HAZ/F82H interface, and segregation of substitutional elements of precipitate were found in the area middle of HAZ, and no precipitates was present in the area $<100\text{ }\mu\text{m}$ away from WM/HAZ interface. Moreover, it was found that the Cr and C concentration in precipitates depended upon the distance from WM/HAZ interface. In the annealed specimens, it was found that the $M_{23}C_6$ gradually decomposed with the temperature increasing from 1073 to 1173 K accompanied with the Cr and C concentration change. The similar feature of Cr segregation observed in the midway-HAZ was confirmed under annealed at 1173 K specimen. The temperature gradient profile in HAZ during fiber laser welding was established by combining data sets in as-welded specimen and annealed specimens. This is the approach for the first time that allows experimental evaluation of the temperature history and distribution upon welding, although this evaluation left a certain error whose reasons will be investigated in the future study.

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