Effect of HIP Temperature and Cooling Rate on Microstructure and Hardness of Joints for ODS-RAFM Steels and JLF-1 Steel*)

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Dissimilar-metal joints between ODS-RAFM (oxide-dispersion-strengthened reduced activation ferritic/martensitic) steels and JLF-1 steel were fabricated by hot isostatic pressing (HIP) at 1000 - 1100 °C with a cooling rate of 5 °C/min. After the HIP, it was always quenched martensite for JLF-1 steel. However, coarse precipitates were found in 9Cr-ODS. Additional annealing experiments to simulate HIP conditions were conducted for 9Cr-ODS with cooling rate ranged from 0.5 to 36 °C/min at 800 - 1100 °C. The results showed that, to form quenched martensite for 9Cr-ODS, the HIP temperature should be above 1000 °C with cooling rate no less than 25 °C/min. When the cooling rate is increased to 36 °C/min, the microstructure of 9Cr-ODS is quenched martensite with precipitate size similar as that before HIP. If the limitation of precipitate size in 9Cr-ODS is 0.2 μm, HIP temperature above 1050 °C with cooling rate no less than 30 °C/min is needed. In this case, post-weld heat treatment (PWHT) with only tempering is necessary to recover the microstructure of 9Cr-ODS to tempered martensite. For 12Cr-ODS, the HIP temperature and cooling rate has no effect on hardness and precipitate size. PWHT is not necessary for the single-metal joint of 12Cr-ODS from the view point of precipitation control. However, for the dissimilar-metal joints between ODS-RAFM steels and JLF-1 steel, the PWHT condition should be comprehensively determined by considering microstructural evolution of each part in the joints after HIP.

Keywords: ODS-RAFM, hot isostatic pressing, coarse precipitate, cooling rate, post-weld heat treatment

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1. Introduction

Because of their excellent high-temperature mechanical properties [1, 2], ODS-RAFM (oxide-dispersion-strengthened reduced activation ferritic/martensitic) steels including 9Cr-ODS and 12Cr-ODS can be utilized partly for the surface of fusion blanket, by bonding with conventional RAFM steels to enhance the acceptable temperature of the blanket surface by 100 - 150 °C. Thus in addition to single-metal bonding of the steels, dissimilar-metal bonding between ODS-RAFM steels and conventional RAFM steels is also essential to the construction of advanced fusion blanket systems applied with ODS-RAFM steels. Hot isostatic pressing (HIP) has been selected as the first candidate method [3] to make large-area and complicated-shape plate bonding for fusion blanket structure. In the previous work [4], dissimilar-metal joints between 9Cr-ODS and JLF-1 were made by HIP at 1000 - 1100 °C under the pressure of 191 MPa for 3 h with a cooling rate of 5 °C/min. The microstructure of JLF-1 base metal is always quenched martensite after the HIP. However, because the cooling rate is too slow, coarse precipitates always existed in the 9Cr-ODS base metal. Post-weld heat treatment (PWHT) with normalization at 1050 °C for 1 h with a fast cooling rate of 36 °C/min can eliminate the coarse precipitates. And then by the following tempering, the microstructure can be recovered to tempered martensite as that before HIP. By considering the fusion blanket fabrication process in the future and saving cost of the PWHT, it is necessary to further investigate the effect of HIP temperature and cooling rate on the microstructure of 9Cr-ODS. 12Cr-ODS is another kind of ODS-RAFM steel with ferrite structure. It is also necessary to investigate the microstructural evolution after HIP for 12Cr-ODS. Proper PWHT conditions are expected to be recommended for well bonding of the above-mentioned steels by considering HIP conditions with different temperatures and cooling rates, from the view point of microstructural control.

2. Materials and Experimental Procedure

Chemical composition of the as-received materials is shown in Table 1. The final heat treatments of the as-received materials were annealing at 1200 °C for 1 h for 12Cr-ODS, normalization at 1050 °C for 1 h for both 9Cr-ODS and JLF-1, followed by tempering at 800 °C for 1 h

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Table 1 Chemical composition of the as-received materials.

Material	W	Cr	Mn	V	Ta	Ti	C	N	Y
9Cr-ODS	1.97	9.08	0.09			0.23	0.14	0.013	0.29
12Cr-ODS	1.90	11.65	0.02			0.29	0.035	0.005	0.18
JLF-1	1.98	9.00	0.49	0.20	0.083		0.09	0.015	

Table 2 Experimental procedure in the present study.

Materials/joints	Temperature (°C)×3h	Cooling rate (°C/min)	Experimental device
9Cr-ODS—JLF-1	1000, 1050, 1100	5	Commercial HIP furnace
9Cr-ODS	800, 900, 1000, 1050, 1100	0.5, 25, 30, 36	Lab-scale image furnace
12Cr-ODS—JLF-1	1050, 1100	5	Commercial HIP furnace
12Cr-ODS	1050	0.5, 25, 30,36	Lab-scale image furnace

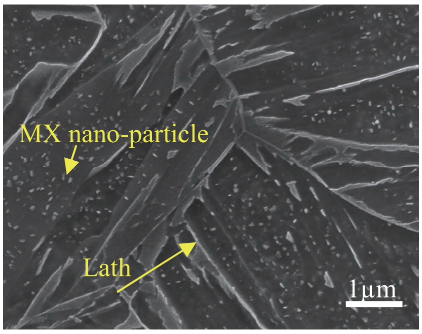

Fig. 1 Microstructure of JLF-1 after HIP (SEM image).

for 9Cr-ODS and at 780 °C for 1 h for JLF-1. The microstructure is tempered martensite for both 9Cr-ODS and JLF-1, and ferrite for 12Cr-ODS. 9Cr-ODS and 12Cr-ODS disks used for HIP were 5 mm in thickness and 24 mm in diameter. JLF-1 blocks were 20 mm in thickness and 24 mm in diameter. For each HIP group, one 9Cr-ODS or 12Cr-ODS disk was sandwiched between two JLF-1 blocks and sealed into a soft steel capsule by using electron beam welding. As shown in Table 2, HIP was carried out in a commercial HIP furnace under a pressure of 191 MPa for 3 h at 1000 °C, 1050 °C, and 1100 °C, respectively. The cooling rate of the HIP was 5 °C/min.

To simulate the HIP conditions and investigate the effect of HIP temperature and cooling rate, as also depicted in Table 2, additional annealing experiments for 9Cr-ODS and 12Cr-ODS sheet specimens with 0.5 mm in thickness, 5 mm in width and length were carried out at different temperature of 800 - 1100 °C for 3 h with cooling rates from 0.5 to 36 °C/min in a lab-scale image furnace in vacuum at pressures less than 5.22×10^{-4} Pa.

Hardness tests were carried out at 300 gf for 30 s for the specimens after HIP or annealing. Microstructural characterization with scanning electron microscopy (SEM) was conducted after etching with picric acid for 30 s for 9Cr-ODS and JLF-1 and 60s for 12Cr-ODS to reveal the microstructure especially precipitates.

3. Results and Discussion

3.1 Hardness and microstructure for JLF-1

JLF-1 is a kind of non-ODS conventional RAFM steel with 9 wt.% Cr. It is well known that, the microstructure for 9Cr RAFM steels [5] should be tempered martensite with nano-particles of MX (M: V, Ta; X: N, C) in the matrix, and precipitates of mainly carbides $M_{23}C_6$ (M: Cr rich) and Laves phase Fe_2W at lath and grain boundaries. In Tan *et al.*'s [6] investigation about 9Cr RAFM steel, the complete decomposition temperature of precipitates $M_{23}C_6$ and Laves phase is below 1000 °C, and that of MX nano-particles (V, Ta)(C,N) is about 1200 °C. In the present study, for JLF-1 after HIP at 1000 - 1100 °C with the cooling rate of 5 °C/min, the hardness is 410 HV, and the microstructure is always full quenched martensite without any precipitates of $M_{23}C_6$ carbides and Laves phase Fe_2W on the lath boundaries, as shown in Fig. 1. The temperature during HIP can dissolve all elements in the precipitates of $M_{23}C_6$ carbides and Laves phase Fe_2W into the matrix, only MX nano-particles remained in the matrix, and the HIP cooling rate of 5 °C/min is enough for JLF-1 to quench. For the single-metal bonding of JLF-1, PWHT with only tempering such as at 780 °C for 1 h is necessary to recover the microstructure to tempered martensite as that before HIP.

3.2 Hardness and microstructure for 9Cr-ODS

Figure 2 shows the hardness of 9Cr-ODS. After HIP at 1000 °C with the cooling rate of 5 °C/min, 9Cr-ODS is softened with the hardness decreased by 10 HV compared to that of the as-received, and the microstructure is ferrite and coarse precipitates with mean size of 0.65 μm [7]. However the higher temperature of 1050 °C and 1100 °C induced quenched martensite for 9Cr-ODS with hardness up to 440 HV. The microstructure is quenched martensite with still smaller coarse precipitates [4]. Additional annealing experiments showed that when the cooling rate is slow as 0.5 °C/min, hardening with quenched martensite was never induced. The microstructure would be always ferrite and coarse precipitates. When the cooling rate is fast with more than 25 °C/min at the HIP temperature above 1000 °C, hardening is induced, and the microstructure would be quenched martensite.

9Cr-ODS is a kind of ODS-RAFM steel with high-density nano-particles. The microstructure for 9Cr-ODS should be tempered martensite with high-density nano-particles of Y-Ti-O compounds in the matrix [1,2] and precipitates of mainly carbides $M_{23}C_6$ and Laves phase Fe_2W on grain boundaries [8, 9]. In the present study, the microstructure of 9Cr-ODS at 1050 °C with different cooling rate is shown in Fig. 3. Coarse precipitates with size of 0.66 μm appear on the grain boundaries when the cooling rate is slow as 0.5 °C/min. When the cooling rate is increased to 5 °C/min and 30 °C/min, the precipitate size decreases to 0.25 μm and 0.18 μm, respectively. And when

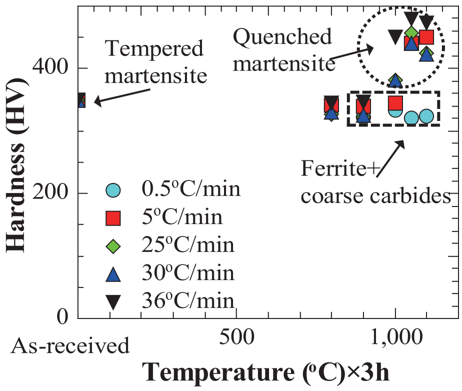

Fig. 2 Effect of temperature and cooling rate on hardness of 9Cr-ODS.

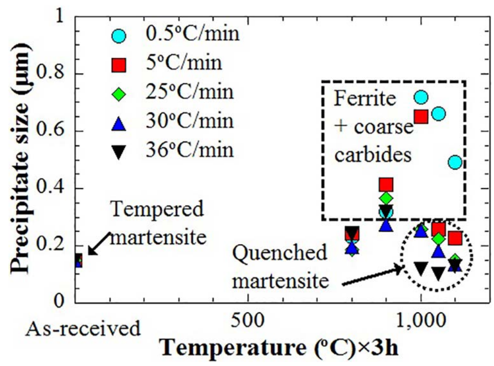

Fig. 4 Effect of temperature and cooling rate on precipitate size in 9Cr-ODS.

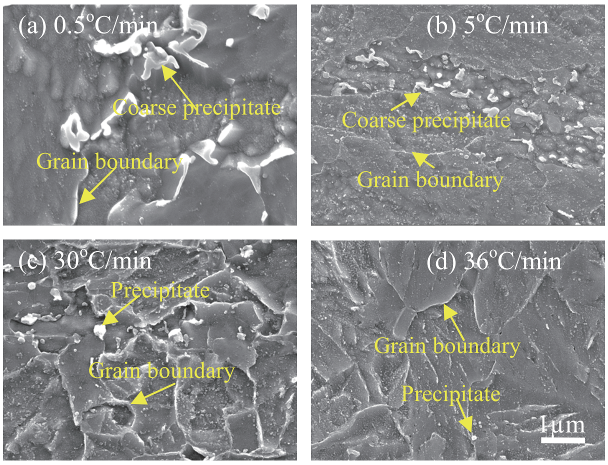

Fig. 3 Microstructure (SEM images) of 9Cr-ODS at 1050 °C for 3 h with different cooling rates, (a) 0.5 °C/min, (b) 5 °C/min (HIP condition), (c) 30 °C/min, (d) 36 °C/min.

the cooling rate is fast as 36 °C/min it is quenched martensite with occasionally normal precipitates with size of only 0.1 μm.

The effect of temperature and cooling rate on precipitate size in 9Cr-ODS is shown in Fig. 4. The precipitate size in the as-received 9Cr-ODS is 0.15 μm [7]. For the HIP at 1000 °C with the cooling rate of 5 °C/min, the precipitate size is 0.65 μm in ferrite structure. When HIP at higher temperature of 1050 °C and 1100 °C the precipitate sizes in the quenched martensitic structure are decreased to 0.26 μm and 0.23 μm, respectively. The additional annealing experiments showed that, when the cooling rate is slow as 0.5 °C/min, there are always coarse precipitates with size 0.5 - 0.75 μm in the ferrite structure. When the cooling rate is fast as larger than 30 °C/min at temperature no less than 1050 °C, the precipitate size is less than 0.2 μm. When the cooling rate is 36 °C/min, the precipitate size can be recovered to the similar level as that in the as-received condition, i.e. 0.15 μm.

During HIP of 9Cr-ODS, decomposition of precipitates of $M_{23}C_6$ carbides and Laves phase Fe_2W may be retarded by the high-density Y-Ti-O nano-particles. The complete decomposition temperature of precipitates is considered to be above 1100 °C. When HIP at 1000 - 1100 °C in the present study, the precipitates cannot be completely decomposed and still partly remained in 9Cr-ODS. If the cooling rate after HIP is fast enough, the remained precipitates cannot be nucleation sites to grow as coarse precipitates. Otherwise, if the cooling rate is too slow, coarse precipitates would be formed.

In the present study, hardness and microstructure of 9Cr-ODS is unstable as the HIP temperature and cooling rate varies. 9Cr-ODS is martensitic steel with the carbon content of 0.14 wt.% as depicted in Table 1. When the HIP temperature is high enough (eg. up to 1050 °C) and the cooling rate is fast enough (eg. up to 36 °C/min), the carbon can be mostly dissolved into the BCT structure to form quenched martensite. However, as the HIP temperature and cooling rate decreased too much (eg. ≤ 1000 °C and ≤ 5 °C/min), quenched martensite cannot be formed, and the carbon can be diffused to make coarser precipitates get out in ferrite structure.

3.3 Hardness and microstructure for 12Cr-ODS

As depicted in Fig. 5, the HIP at 1050 °C and 1100 °C with the cooling rate of 5 °C/min has no effect on hardness of 12Cr-ODS. The additional annealing experiments at 1050 °C with different cooling rate also have no effect on hardness evolution. The hardness is always 310 HV as that for the as-received.

Li *et al.* [10] reported that, besides high-density Y-Ti-O nano-particles in the ferrite matrix of 12Cr-ODS, precipitates with size of 0.279 ± 0.05 μm of TiC_xO_{1-x} and $M_{23}C_6$ were also detected by X-ray diffraction. Figure 6 shows the microstructure of 12Cr-ODS at 1050 °C with different cooling rate. The mean size of precipitates is stable with less than 0.2 μm as depicted in Fig. 7, same as that in the as-received.

As shown in Table 1, the carbon content is only 0.035 wt.% in 12Cr-ODS. It is known from the Fe-C phase diagram that, the maximum carbon solubility is 0.022 wt.%

in the ferrite structure [11]. The extra carbon of about 0.013 wt.% in content is believed to be already fully precipitated out as $M_{23}C_6$ carbides and TiC_xO_{1-x} compounds during the fabrication process of 12Cr-ODS. No extra carbon can be precipitated out again during the HIP or the annealing experiments. Thus the microstructure of 12Cr-ODS is stable.

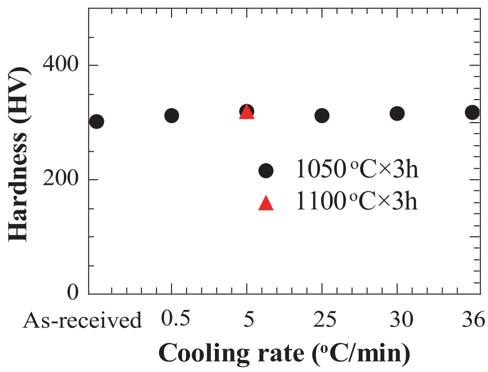

Fig. 5 Effect of temperature and cooling rate on hardness of 12Cr-ODS.

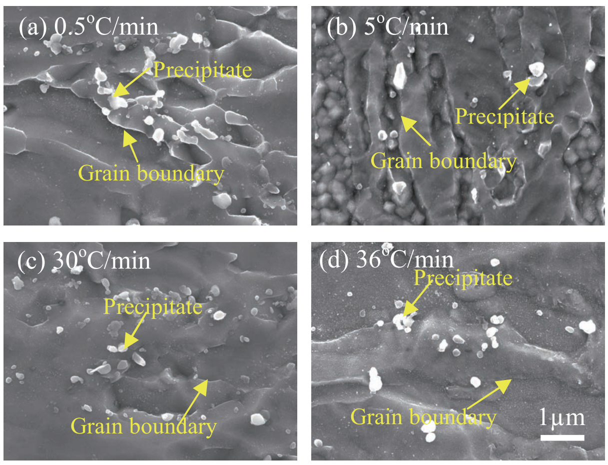

Fig. 6 Microstructure (SEM images) of 12Cr-ODS at 1050 °C for 3 h with different cooling rates, (a) 0.5 °C/min, (b) 5 °C/min (HIP condition), (c) 30 °C/min, (d) 36 °C/min.

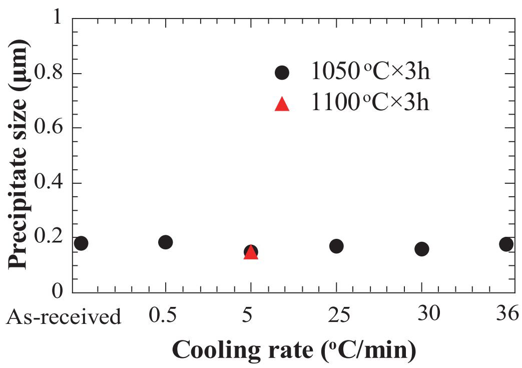

Fig. 7 Effect of temperature and cooling rate on precipitate size of 12Cr-ODS.

3.4 Proposed HIP and PWHT conditions

In the previous study [4], 1000 °C for HIP is too low to bond well the joint between 9Cr-ODS and JLF-1. Since the joint fractured at the interface with about one third of the yield strength of 9Cr-ODS [4], there may be spherical unbounded areas at the interface when HIP at low temperature of 1000 °C. Therefore the HIP temperature should be no less than 1050 °C for better bonding of RAFM steels. The high temperature during HIP have changed the microstructure of 9Cr-ODS and JLF-1. The joints after HIP cannot be applied in the fusion blanket directly, since quenched martensite is hard and brittle; coarse precipitates not only degrade strength, but also can be crack initiations [5] during the long-term application of the joints. Therefore, PWHT is necessary to recover the microstructure of them to tempered martensite with normal size of precipitates of carbides $M_{23}C_6$ and Laves phase Fe_2W. If the limitation of precipitate size is 0.2 μm, the HIP cooling rate should be no less than 30 °C/min for 9Cr-ODS to form quenched martensite. In this case, only tempering is needed in the following PWHT procedure. By considering the fusion blanket fabrication in the future, Table 3 proposed the PWHT of the joints according to different HIP conditions from the view point of microstructural control. For the single-metal bonding of JLF-1, because it is full quenched martensite after HIP, only tempering is necessary for the PWHT to recover the microstructure to tempered martensite. For single-metal bonding of 9Cr-ODS, if the cooling rate during HIP is less than 30 °C/min, PWHT with normalization is necessary to dissolve the elements in coarse precipitates into matrix, and then followed by tempering the microstructure can be recovered to tempered martensite with normal size of precipitates. If HIP cooling rate of 9Cr-ODS more than 30 °C/min, only PWHT with tempering is necessary. For single-metal bonding of 12Cr-ODS, since the HIP cooling rate has no effect on microstructure and hardness, PWHT is not necessary in this case. For the dissimilar-metal bonding between 9Cr-ODS and JLF-1, the PWHT condition should be conducted by considering the microstructural evolution of the 9Cr-ODS side. For the dissimilar-metal bonding between 12Cr-ODS and JLF-1, microstructure recovery of JLF-1 side should be noted. However, more mechanical property evalua-

Table 3 Proposed PWHT according to HIP conditions.

Joints	HIP		PWHT	
	Temperature (°C)	Cooling rate (°C/min)	Temperature (°C)	Cooling rate (°C/min)
JLF-1—JLF-1	1050-1100	≥5	780	≥5
9Cr-ODS—9Cr-ODS	1050-1100	≤30 ≥30	1050+800 800	≥30 ≥30
12Cr-ODS—12Cr-ODS	1050-1200	≥0.5	Not necessary	Not necessary
9Cr-ODS—JLF-1	1050-1100	≤30 ≥30	1050+780 780	≥30 ≥30
12Cr-ODS—JLF-1	1050-1100	≥5	780	≥5

tion for the above-mentioned joints by tensile tests, fatigue tests, and creep tests should be carried out to choose proper HIP and PWHT conditions.

4. Conclusions

The microstructure for JLF-1 is always quenched martensite after HIP with a cooling rate of 5 °C/min. However coarse precipitates appeared for 9Cr-ODS. Annealing experiments to simulate HIP temperature and cooling rate was studied for 9Cr-ODS and 12Cr-ODS. The microstructure can be ferrite and coarse precipitates, quenched martensite and smaller coarse precipitates, and quenched martensite without coarse precipitates for 9Cr-ODS at different temperatures with different cooling rates. Thus different kind of PWHT with normalization and tempering or only tempering should be considered to recover microstructure of 9Cr-ODS to tempered martensite with normal size of precipitates. For 12Cr-ODS, HIP temperature and cooling rate has no effect on hardness and microstructure, therefore PWHT is not necessary for the single-metal bonding of it from the view point of precipitation control. However, for the dissimilar-metal bondings between ODS-RAFM steels and JLF-1, the PWHT should be considered carefully by evaluating the microstructure of both sides of the joints after HIP.

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