§16. Effect of HIP Temperature and Cooling Rate on Microstructure of 9Cr-ODS in Joints

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(Fe-9.08Cr-0.14C-1.97W-0.23Ti-0.29Y-9Cr-ODS is a kind of reduced 0.16O-0.013N) activation ferritic/martensitic (RAFM) steel with high-density Y-Ti-O nano-oxide particles. It has more excellent high-temperature mechanical properties than conventional RAFM steels such as JLF-1 and F82H. Thus 9Cr-ODS can be used partly for the surface of fusion blanket by bonding with conventional RAFM steels. In the previous work, Hot isostatic pressing (HIP) at 1000-1100°C under a pressure of 191MPa for 3h has been carried out for the dissimilar-metal bonding between 9Cr-ODS and JLF-1.11 HIP always induced quenched martensite for JLF-1. Therefore, if for the singlemetal bonding for JLF-1, post-weld heat treatment (PWHT) with only tempering is needed to recover its microstructure to the original tempered martensite. However, for the joints including 9Cr-ODS, because the cooling rate after HIP (5°C/min) is too slow to form full quenched martensite for 9Cr-ODS, coarse carbides always induced which may be crack initiation in the future application. In addition, HIP at low temperature of 1000°C produced ferrite structure with coarse carbides.<sup>2)</sup> This microstructure caused softening of 9Cr-ODS. The purpose of the present study is to fully investigate effect of HIP temperature and cooling rate on microstructure of 9Cr-ODS, to seek proper PWHT condition by considering fusion blanket fabrication in the future.

To simulate HIP condition and investigate the effect of HIP temperature and cooling rate, annealing experiments were carried out at different temperature of 800-1100°C for 3h with cooling rates from  $0.5^{\circ}$ C to  $36^{\circ}$ C/min in a lab-scale image furnace in vacuum at pressures less than  $5.22 \times 10^{-4}$  Pa.

Figure 1 (a) shows the hardness of 9Cr-ODS. When the HIP cooling rate is slow as  $0.5^{\circ}$ 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  $25^{\circ}$ C/min at the HIP temperature above  $1000^{\circ}$ C, hardening is induced, and the microstructure would be quenched martensite.

The microstructure for 9Cr-ODS should be tempered martensite with high-density nano-particles of Y-Ti-O compounds in the matrix and precipitates of mainly carbides  $M_{23}C_6$  and Laves phase Fe<sub>2</sub>W on grain boundaries.<sup>3-5)</sup> Temperature and cooling rate have obvious effect on precipitate size in 9Cr-ODS, as shown in Fig.1 (b). The precipitate size in the as-received 9Cr-ODS is 0.15  $\mu$ m.<sup>2)</sup> When the cooling rate is slow as 0.5°C/min, there are always coarse precipitates with size 0.5-0.75 $\mu$ m in the ferrite

structure. When the cooling rate is fast as larger than  $30^{\circ}$ C/min at temperature no less than  $1050^{\circ}$ C, the precipitate size is less than  $0.2\mu$ m. When the cooling rate is  $36^{\circ}$ C/min, the precipitate size can be recovered to the similar level as that in the as-received condition, i.e.  $0.15\mu$ m. During HIP of 9Cr-ODS, decomposition of precipitates of M<sub>23</sub>C<sub>6</sub> carbides and Laves phase Fe<sub>2</sub>W 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 below 1100°C, 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. Otherwise, coarse precipitates would form.



Fig. 1. Effect of HIP temperature and cooling rate on (a) hardness and (b) precipitate size of 9Cr-ODS.

In the present study, hardness and microstructure of 9Cr-ODS is unstable as the HIP temperature and cooling rate varies. 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 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<sub>23</sub>C<sub>6</sub> and Laves phase Fe<sub>2</sub>W. If the limitation of precipitate size is 0.2µm, 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. 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.

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