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Effect of Surface Preparation on CLAM/CLAM Hot Isostatic Pressing diffusion bonding joints

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ABSTRACT

Surface preparation is essential for the Hot Isostatic Pressing (HIP) diffusion bonding of RAFM steels. Hot Isostatic Pressing (HIP) diffusion bonding experiments on China Low Activation Martensitic (CLAM) steel was performed to study the effect of surface preparation. A few approaches such as hand lapping, dry-milling and grinding etc., were used to prepare the faying surfaces of the HIP joints. Different sealing techniques were used as well. The HIP parameters were 150 MPa/3 h/1150 °C. After post HIP heat treatment (PHHT), the tensile and Charpy impact tests were carried out. The results showed that hand lapping was not suitable to prepare the faying surfaces of HIP diffusion bonding specimens although the surface roughness by hand lapping was very low.

1. Introduction

Structural materials of fusion blankets are subjected to high energy and high flux neutron irradiation, high thermal and mechanical loads. Reduced activation ferritic/martensitic steels are the leading candidate structural materials for the blanket and first wall of DEMO reactors for many advantages. The CLAM (China Low Activation Martensitic) steel is under development in the Institute of Plasma Physics, Chinese Academy of Sciences (ASIPP) under collaboration with many other universities and institutes [1,2]. It is the candidate structural material in the design of FDS (Fusion Design Study) series liquid LiPb blanket fusion reactors [3–6] and the Dual-Functional Lithium Lead Test Blanket Module (DFLL-TBM) [7,8] for International Thermonuclear Experimental Reactor (ITER), which are also being carried out in ASIPP. In parallel with properties study on CLAM, fabrication technologies development is being conducted as well in order to provide technical basis for TBM in the ITER.

The first wall, the cooling and the stiffening plates in the fusion reactor blanket concepts are provided with complex arrays of helium cooling channels in order to meet thermohydraulic requirements. Solid Hot Isostatic Pressing (HIP) bonding is expected to provide the most promising fabrication method for these complex structures which needs to withstand the severe condition in the fusion reactor [9]. Thus, the research of the HIP bonding technique on CLAM/CLAM is being carried out.

Surface preparation is essential for the HIP diffusion bonding of RAFM steel. So the faying surfaces of HIP diffusion bonding specimens were prepared in several ways in the recent experiments. The results and discussion are given in the following sections.

2. Experimental setup

The HIP specimens of CLAM were taken from a long bar with 45 mm in diameter. The specimen sizes were 40 mm in diameter and 40 mm in length. The chemical composition for the material is shown in Table 1. Heat treatment of the bar was 980 °C/30 min/air, 760 °C/90 min/air.

Hand lapping, grinding and dry turning were used to prepare the joining surface of the specimens in order to study the surface preparation's influence on the properties of the HIP joints. The hand lapping procedure is as follows. First, the specimen is ground by grinding machine. Second, the specimen surface is hand lapped on a large flat plate. Then the surface of the specimen was very smooth and its surface roughness was very low.

The specimens were washed in an ultrasonic bath with mixed solution of acetone and ethanol. Then they were outgassed at high temperature in a vacuum furnace for several hours. After that, several pairs of specimens were directly sealed at the edge of the faying surfaces by Electron Beam Welding (EBW). The others were canned in capsules made of stainless steel 304 l. The capsules were outgassed for tens of hours under about 300 °C, after which they were cooled down to room temperature and sealed. The temperature, pressure and time of the HIP were 1150 °C, 150 MPa and 3 h, respectively. The post HIP heat treatment (PHHT) was 950 °C/60 min/air and 760 °C/90 min/air to regain the microstructure.

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Table 1
Chemical compositions of CLAM (HEAT 0603A) (wt%)

Elements	Cr	W	V	Ta	Mn	C	Si
wt%	9.23	1.37	0.20	0.15	0.43	0.15	0.083
Elements	O	N	S	Cu	Al	Ti	Ni
wt%	0.0017	0.00585	0.0042	0.010	0.027	0.0032	0.11

3. Results and discussion

3.1. Tensile tests

The tensile specimens with gage section of ϕ 5 × 25 mm were cut down from the joints and the joining interfaces were at the middle of gage section. The tensile properties of the joints and those of base metal are shown in Table 2.

3.2. Microstructure observation

The testing pieces for metallurgical observation were taken at the interface of the joints. They were wet ground with grits up to #1200, polished by buffing with 1 μm diamond powder, followed by etching. The microstructure of the joint was observed through optical microscope. Micrographs of 4# and 5# joints are shown in Fig. 1. The grain number of the specimens was evaluated. The ASTM grain size number of base CLAM steel under standard heat treatment is about 11. After the HIP and PHHT courses, the number is about 4.

TaC is the most stable carbide in CLAM steel. The grain coarsening in the HIP course is believed to proceed due to TaC dissolution at grain boundaries [10]. Tamura et al. found that the solubility of TaC in F82H steel could be calculated by the following equation [11]:

$$\log\{[\%\mathrm{Ta}][\%\mathrm{C}]\} = -7027/T + 3.16,$$

where T is the temperature in K and the contents for Ta and C are in mass percentage. The composition of CLAM steel is close to that of F82H steel except a little difference in W and Ta contents, so the TaC solubility can be calculated roughly according to the equation. According to the calculation, Ta in CLAM steel is almost dissolved totally at about 1150 °C, which may cause significant grain growth. Although, PHHT was carried out, the grain of the specimens did not recover to the original size, which shows that the HIP temperature needs to be optimised.

Table 3
Impact absorbed energy of the HIP joints.

No.	Temperature (°C)	Impact absorbed energy (J)
1#	20	10.3
2#	20	34.4
3#	20	89.6
4#	20	8.4
5#	20	87.4

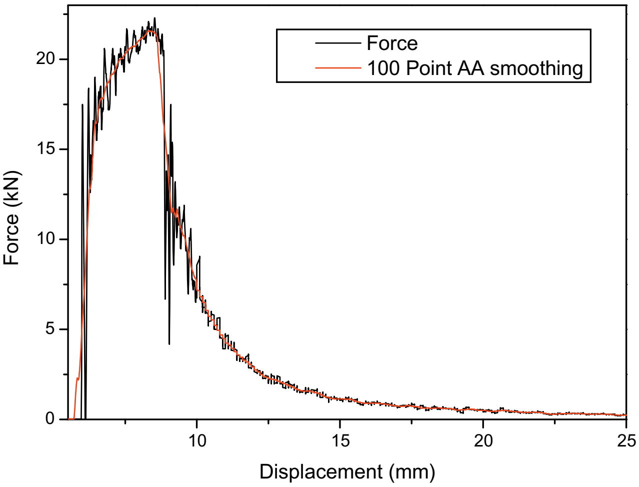

Fig. 2. Force vs. displacement during the impact test of 5#.

3.3. Charpy impact test (CIT)

When the blanket is exposed to some related events, e.g. plasma disruption, the FW and stiffening plate will suffer from shock loading [12]. So the CIT for the joints of CLAM/CLAM needs to be done

Table 2
Tensile properties of the joints and base metal.

No.	Surface preparation	Surface roughness (μm)	Sealing method	Ultimate tensile strength (MPa)	Area reduction (%)
1#	Hand lapping	0.16	Capsule	704.4	77.0
2#	Hand lapping	0.16	Capsule	716.2	73.0
3#	Grinding	0.63	Capsule	730.0	73.4
4#	Hand lapping	0.16	EB welding	741.2	77.0
5#	Dry milling	1.25	EB welding	733.5	75.1
Base metal				751.5	72.5

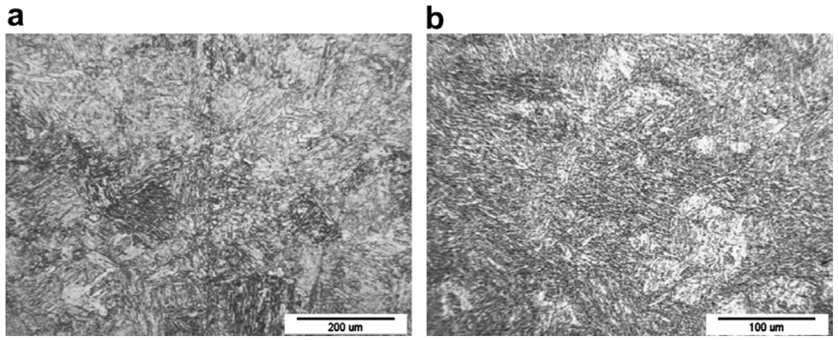

Fig. 1. Metallurgical observation of HIP joints at the interface by optical microscope, (a) 4# and (b) 5#.

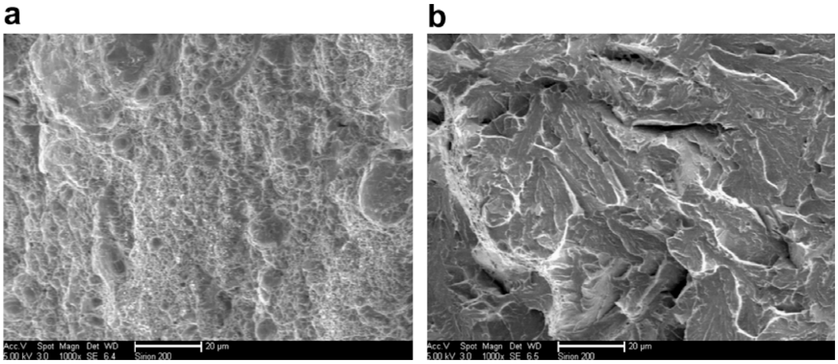

Fig. 3. CIT specimen fractograph of 5# by SEM, (a) near the notch and (b) far from the notch.

to qualify the joints further. And the HIP diffusion bonding experiments on the other RAFMs showed that CIT results were sensitive to properties of the joints [9,13].

Standard U-shape CIT specimens, i.e. with the sizes of $10 * 10 * 55\ \mathrm{mm}^3$, were used and the notches were made just at the interface. The CITs were performed on impact testing machine with oscillometer (KPR450) produced by Zwick company. The Impact Absorbed Energy (IAE) values are listed in Table 3. The absorbed energy of base metal at room temperature is larger than 250 J [14,15].

Table 3 shows that the tensile properties of the joints were identical to that of base metal, but the absorbed energies of all joints were low, especially for the specimens prepared with hand lapping. The IAE value of 3# and 5# is around 35% of that of the base metal. The force vs. displacement curve of specimens from 5# during the impact test is as shown in Fig. 2, which indicates that the fracture mode of the specimens is brittle. When the force reached the maximum, the crack began to expand quickly.

Many dimples could be found from the CIT specimen fractographs of 5# as shown in Fig. 3. These indicate that the fracture mode is ductile at the beginning of the crack. However, this area is rather small, and the river pattern in Fig. 3(b) shows the crack is mainly cleavage fracture.

3.4. Discussion

As stated above, the tensile properties of the joints are good enough, but the impact properties under room temperature are still poor, especially for the joint prepared with hand lapping. This phenomenon indicates that the hand lapping is not suitable to be used in the HIP diffusion bonding of CLAM. The possible reason for that could be explained as follows. The surface of the specimen after hand lapping was gray, which indicated that there was a thick oxide layer. And solution and spheroidization of the oxide layer is very important to quality of the diffusion bonding joint [16]. And required time for the solution of the oxide is proportional to square of thickness. As for the insoluble oxide, e.g. alumina and silica, the breaking and spheroidization of oxide film is needed. However, the quite smooth surface with very low roughness by hand lapping makes the process difficult. So the thick oxide layer is the possible reason for low impact absorbed energy. Also, this experimental result agrees with the opinion of Cardella et al. [13] that properties of the diffusion bonding joints mainly correlated to the cleanness of the faying surface but not the surface roughness.

Although, the bonding line for 5# is almost invisible as shown in Fig. 1(b), IAE values of 3# and 5# by grinding and dry-milling are only close to 90 J, and the fractograph shows mainly cleavage fracture at room temperature. This may be caused by over coarse grain due to the high outgassing temperature in the furnace and the HIP temperature, which indicates these parameters need to be optimized.

4. Conclusions

HIP diffusion bonding experiments on CLAM steel were performed to study the influence of surface preparation on the joints. Main conclusions can be summarized as follows:

1. The grains of CLAM steel after HIP and PHHT were over coarsened, because 1150 °C is too high for CLAM HIP diffusion bonding, and the effect PHHT on grain size recovery is limited.
2. The hand lapping is not suitable for HIP diffusion bonding of CLAM, and the properties of the joints is mainly correlated to the surface cleanness not the surface roughness.

Further experiments to optimise the HIP diffusion bonding technique of CLAM will be performed. Fabrication of small first wall modules is also planned.

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