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Preliminary experiment on compatibility of SiC_f/SiC composites in static liquid LiPb at 700 °C

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ABSTRACT

SiC_f/SiC composite is considered as one of the most promising structural material candidates for fusion reactors. Its corrosion behavior in liquid LiPb at high temperature needs to be investigated in details. A capsule named DRAGON-ST was designed and built for static corrosion experiment at 700 °C as a first step in order to evaluate the corrosion behavior of homebred SiC_f/SiC composite with LiPb, it was characterized by weight change and observation of corroded surfaces. The results after 500 h experiment showed that the 3D SiC_f/SiC composite with CVD (chemical vapor deposition)–SiC coating was stable in the liquid LiPb, most area of the composites surface and the matrix remained well. Further study needs to be carried out to evaluate the compatibility of SiC_f/SiC composites with LiPb completely.

1. Introduction

LiPb blanket design is the one of the promising choice for fusion reactor blankets. In China, a series of FDS fusion reactors and their LiPb blankets have been designed for years [1–6]. The structure material of blankets is CLAM (China Low Activation Martensitic) [7–11], and liquid metal LiPb is considered as tritium breeder, neutron multiplier and coolant. Among them, the outlet temperature of LiPb for the DLL (dual-coolant lithium-lead) blanket is 700 °C.

SiC fiber-reinforced SiC matrix composites (SiC_f/SiC) is being considered as a candidate structural material for fusion reactors because of their low-induced radioactivity by 14 MeV neutron irradiation, their excellent high-temperature strength, and corrosion resistance, etc. [12]. There are three general fusion reactor design concepts using SiC_f/SiC composites, such as ARIES-AT [13] designed by USA, DREAM [14] designed in Japan and TAURO [15] in the EU. In addition, SiC_f/SiC is designed as one of the candidate materials for FCI (flow channel inserts) inside the LiPb coolant channel and manifold to keep the temperature of the structural material at moderate temperature [1,16]. Up to now, great progress has been achieved on the development of SiC_f/SiC composites in the world [15,17–21], especially for the fabrication technology and properties optimization.

Because of the urgent deadline for ITER construction, it is severely needed to study on the compatibility of SiC_f/SiC composites with LiPb at high temperature for liquid metal blankets [22–25]. Some work was done in the world. Raffray et al. indicated that SiC_f/SiC composites were practically inert in LiPb at 800 °C for 1500 h in 2001 [14]. Barbier et al. reported, in 2002 [22], that SiC_f/SiC composites did not react with liquid LiPb at 800 °C for 3000 h, it should be stable and compatible under this environment. Pint et al. concluded that monolithic SiC specimens exposed to LiPb for 1000 h at 800 °C and 1100 °C showed no mass change after cleaning in 2006 [25], and suggested that SiC may be limited to <1100 °C in LiPb in 2007 [24], and so on.

In China, a capsule named DRAGON-ST was built for the high-temperature static corrosion experiment, the preliminary experiment was carried out firstly to investigate the feasibility of SiC_f/SiC composites under static LiPb at 700 °C for 500 h.

2. Experimental procedures

2.1. Features of DRAGON-ST

The environment is very critical for this kind of high-temperature static experiment [16,26] because the specimens should be easy to place into the capsule before experiment and

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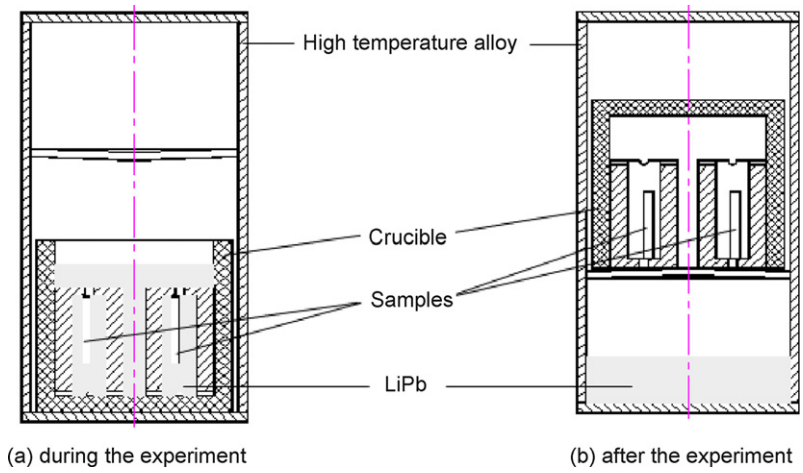

Fig. 1. The scheme of DRAGON-ST. (a) During the experiment; (b) after the experiment.

sampling after experiment, in addition to the material of capsule is required to work well at high temperature. A capsule named DRAGON-ST was built with a monolithic SiC crucible with CVD (chemical vapor deposition) SiC coating inside its surface to contain the LiPb alloy, and mantles made of Mo was used to keep the specimens inside them in order to avoid specimens exposing to the above surface of liquid metal LiPb due to the buoyancy of their different densities. The last process of assembling the capsule was to weld the outer connection by gas-tungsten-arc method.

Firstly, the mantles and specimens, placed into the SiC crucible, were immersed into liquid LiPb inside the sealed capsule, as shown in Fig. 1a, the above atmosphere was pure Ar. After experiment, the capsule is rotated upside down slowly, as shown in Fig. 1b, then the liquid LiPb drains to the bottom of capsule which made the specimens expose to the argon gas atmosphere with little LiPb left on their surfaces. When the capsule cools down to the room temperature, the capsule is cut into two parts to take out the specimens.

2.2. Specimens and corrosion experiment

SiC_f/SiC composites are manufactured by polymer infiltration and pyrolysis (PIP) process. The KD-1 type SiC fiber is covered with CVD–SiC layer which thickness is controlled to about 0.7 μm. The fiber perform has a total fiber volume fraction of about 50%, the chemical composition (wt.%) of the composite is 47.52 Si–48.41 C–4.07 O.

After fabrication, the specimens are cut into pieces with decided dimensions, and no CVD–SiC coating in their cross sections. The dimensions (length × width × height) are 31.1 mm × 2.46 mm × 4.78 mm (marked specimen 1#), 31.4 mm × 4.94 mm × 4.66 mm (marked specimen 2#) and 31.8 mm × 4.92 mm × 4.68 mm (marked specimen 3#), respectively.

When 500 h experiment at 700 °C finished, the specimens are taken out of the capsule and then washed with the cleaning procedure until their weight change remains constant. Unfortunately, a narrow crack is formed in the SiC crucible and did not used any more in the later experiment, it maybe the effect of mechanical vibration during the process of cutting the capsule to take out of specimens.

3. Results

Considering the effect of rough surface, the dimension change was not obtained accurately. The experiment results were characterized by weight changes, observation of corroded surfaces.

Table 1
Specifications of weight change after 500 h exposure.

Specimens no.	Before experi. (g)	After experi. (g)	Weight change	
			(g)	(mg/cm^2)
1#	0.7154	0.9038	0.1884	39.8
2#	1.3785	2.0306	0.6521	100.49
3#	1.3781	1.6568	0.2787	42.4

The metallographic analysis was performed by scanning electron microscope (SEM), and the chemical composites of the corroded layers were evaluated preliminarily by means of energy dispersive X-ray spectroscopy (EDX).

3.1. Weight change

The weight change of specimens before and after experiment was listed in Table 1. It was clear that the weight changed obviously.

Before experiment, the dimension of specimen 2# was similar to that of specimen 3# and was about twice in width than that of specimen 1#. However, after the experiment, the mass increase of specimen 3# was similar with specimen 1# and near to 40 mg/cm^2. However, the mass increase of specimen 2# was more than twice that of the two other specimens.

3.2. Surface investigation

The morphologies of the specimens' surface by SEM and the line scan of EDX were shown in Fig. 2. The surfaces kept almost unchanged. C and Pb content varied almost near to 0 scale line, but Si content varied from 0 to 500 along the whole scanning line and O from 0 to 400 at some local place.

In addition, after removed the covered CVD–SiC coating, the matrix of composite was exposed. The surface observation with SEM and EDX were shown in Fig. 3, the whole surface remained fresh without any corrosion.

4. Discussion

From the maps of surface investigation after the 500 h experiment, most area of the composites surface remained well.

As to the reason of mass increases and different elements content for the corrosion layer, it may be the reason of some influence factors. Each specimen was prepared with two cross sections, oxidation occurred to the specimens' surface that may be caused a

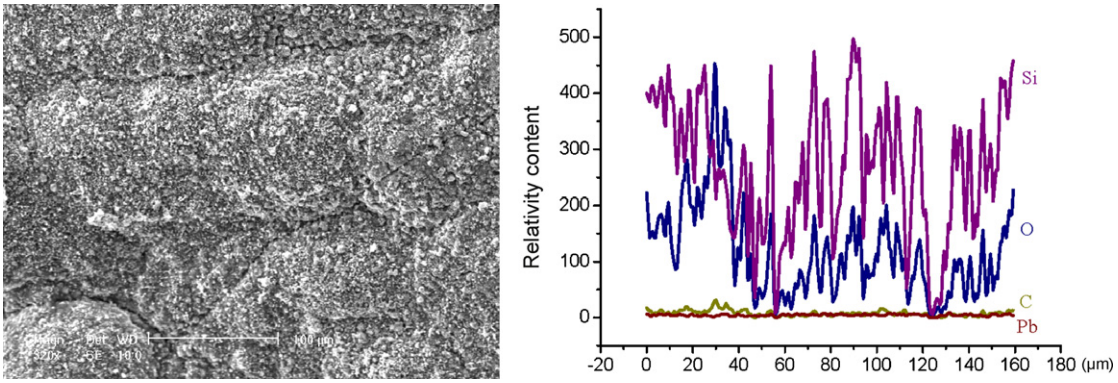

Fig. 2. SEM of specimen surface after 500 h exposure.

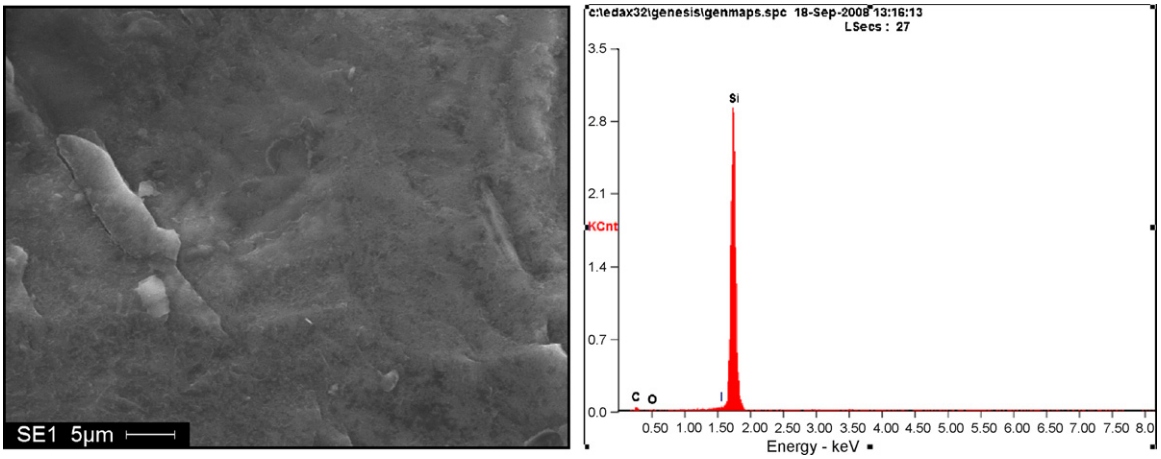

Fig. 3. Surface observation and content test of matrix.

mass increases varied large scale for O content. After hundreds of hours experiment, maybe the penetration of LiPb occurred into the open porosities along the cross section sides, and the weight change increased, it would be the reason for specimen 2# why Pb content was appeared in the EDX map and made the mass increases nearly two times than others. In addition, the experiment process was completed inside the SiC crucible, mass transfer of Si from the SiC crucible occurred that made Si deposited on the surface of composites. Therefore, under the overall effect of O, Si and Pb, mass increase was clearly for the three specimens after experiment.

5. Summary

With the rapid development of fusion reactor, it is time to speed up the development of promising candidate structure material such as SiC_f/SiC composite to fit the requirement of liquid metal LiPb blanket, so is the compatibility experiment. Preliminary exposure experiment on the 3D SiC_f/SiC composite made in China showed good compatibility with static LiPb at 700 °C, the same to the results in the previous paper, most area of the composites surface and the matrix remained well after experiment. But more work is needed urgently to investigate the integrated performance of 3D SiC_f/SiC composite in the later, such as:

(1) The measurements of LiPb environment-induced composite changes after experiment to proof the corrosion behavior of matrix.
(2) Investigation on the cross section of matrix.
(3) Long time experiment is critical and short of in this field at present.
(4) The mechanic property after experiment, and so on.

In addition, the experiment at 700 °C for 1000 h is going on, and it is also needed much longer experience to carry out series of experiments and researches for 3D SiC_f/SiC composite with CVD–SiC.

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