COMPATIBILITY OF CVD SIC WITH Pb-Li AT 800°-1200°C—B. A. Pint and J. L. Moser (Oak Ridge National Laboratory)

OBJECTIVE

The objective of this task is to assess the long-term, high-temperature compatibility of SiC/SiC composites and Pb-Li. One proposed fusion reactor concept uses SiC/SiC composites with a self-cooled Pb-17Li blanket. Another concept uses a SiC/SiC flow channel insert with a dual coolant of He and Pb-Li at 800°C. One attractive feature of ceramic composites is their high temperature capabilities (≥1000°C). However, there is limited compatibility data above 800°C for this system. As the first step in the evaluation process, monolithic SiC is being exposed to Pb-17Li in capsule tests at 800°-1200°C.

SUMMARY

The Pb-Li chemistry was analyzed after the first series of static Pb-17Li capsule tests with monolithic SiC specimens. No Si was detected after the 1000h tests at 800° and 1100°C with a detectability limit of 30ppma (5ppmw). These results confirm the prior results of no specimen mass change and provide a promising but still incomplete picture of compatibility in this system. A second series of longer-term capsule tests is underway to examine potential changes in wetting behavior. A 1200°C capsule test also is being planned.

PROGRESS AND STATUS

Introduction

Among the proposed fusion reactor concepts, silicon carbide composites are a structural material option that is thought to allow the highest reactor operating temperature (1000°-1100°C) and thus the highest operating efficiency.[1,2] Both the TAURO and ARIES-AT proposals have Pb-17at.%Li self-cooled blankets which are attractive because the Pb-17Li acts as coolant, neutron multiplier and tritium breeder.[3] (The eutectic Pb-Li composition was chosen because it has a low melting point of 235°C.) Another concept uses SiC/SiC composite flow channel inserts with a dual coolant blanket based on ARIES-ST.[4] The insert serves as an electrical and thermal insulator between Pb-Li at 800°C and He-cooled ferritic steels. Present assessments of the materials feasibility for both of these strategies are incomplete because there is little information available on the high temperature compatibility of SiC with Pb-Li at temperatures of 800°C and higher. In a static exposure at 427°C, SiC composites (but not monolithic SiC) dissolved in unalloyed Li.[5,6] However, the activity of Li in Pb-17Li is greatly reduced (1.2×10^{-4} at 500°C)[7] suggesting that compatibility would be better with the eutectic.

Prior to the start of this task, results at 800°C had shown limited reaction between Pb-17Li and SiC composites.[8-10] An initial series of static Pb-17Li capsule tests were performed on high-purity (99.9995%) CVD β-SiC specimens and the experimental procedure and results are detailed elsewhere.[11,12] (These experiments used monolithic SiC specimens instead of composites as a first step towards evaluating SiC compatibility to avoid the microstructural and microchemistry complexities of composites and the better compatibility found for monolithic material compared to SiC/SiC composites in Li.[5]) To avoid unwanted reactions of the capsule with Pb-Li, the SiC specimens were contained in SiC capsules. After 1000h at 800°C, no wetting was observed between Pb-Li and SiC and therefore no chemical attack would be expected. At 1100°C, there was evidence of only limited wetting after 1000h. After cleaning the specimens, no mass change was measurable at either temperature suggesting that SiC is compatible with static Pb-17Li to at least 1100°C. To follow up on those observations, the Pb-Li was sent for chemical analysis after the test and the starting Pb material also was analyzed.

Results

The Pb-Li was analyzed after both capsule tests (800° and 1100°C) as was the starting Pb, Table 1. (The variation in Li may be an experimental error. It is magnified by the conversion from mass%.) In Pb-Li, the Si detectability limit was 30ppma (5ppmw) and none was detected. This is significantly less Si than was detected by microprobe analysis (350ppmw) after an 800°C exposure by Kleykamp.[9] The major impurities detected were C and O with low levels of some metals, Table 1. These results suggest that monolithic, high-purity SiC is compatible with Pb-17Li to at least 1100°C in a static environment. More interaction may be expected with the SiC fibers or the fiber-matrix interface. However, it is anticipated that a dense CVD SiC seal coat will cover the outer layer of any SiC composite components.

Long-term capsule tests on monolithic SiC are currently underway at 800°C (5,000h) and 1100°C (2,000h) using the same experimental procedure. Longer times are being investigated because of the limited amount of wetting observed in the 1,000h tests. If wetting is due to a native oxide on the SiC samples, longer times may break down this thin surface layer. Observations with stainless steel and mercury indicate that there can be an extended incubation period to develop wetting.[13] A 1200°C test also is being planned. Welding and oxidation studies are being conducted on alloy 602CA (Ni-26at.%Cr-5Al) to verify its applicability as an outer capsule for this very high temperature.

Future work will eventually need to include flowing liquid metal experiments with a temperature gradient. Static capsule experiments can only be expected to produce a limited picture of the compatibility issue where saturation of one or more of the dissolving components can inhibit further reaction. However, the observation of no detectable dissolved Si in the Pb-Li following exposure is a promising result about the compatibility in this system.

References

[1] A. Hasegawa, A. Kohyama, R. H. Jones, L. L. Snead, B. Riccardi, and P. Fenici, J. Nucl. Mater. 283-287 (2000) 128.
[2] R. H. Jones, L. Giancarli, A. Hasegawa, Y. Katoh, A. Kohyama, B. Riccardi, L. L. Snead, and W. J. Weber, J. Nucl. Mater. 307-311 (2002) 1057.
[3] A. R. Raffray et al., Fusion Eng. Des. 55 (2001) 55.
[4] P. Noajitra, L. Buhler, U. Fischer, S. Malang, G. Reimann, and H. Schnauder, Fusion Eng. Des. 61-62 (2002) 449.
[5] T. Terai, T. Yoneoka, and S. Tanaka (unpublished research from reference #2, 2000).
[6] T. Yoneoka, S. Tanaka, and T. Terai, Mater. Trans. 42 (2001) 1019.
[7] P. Hubberstey, J. Nucl. Mater. 247 (1997) 208.
[8] F. Barbier, Ph. Deloffre, and A. Terlain, J. Nucl. Mater. 307-311 (2002) 1351.
[9] H. Kleykamp, J. Nucl. Mater. 321 (2003) 170.
[10] H. Kleykamp, J. Nucl. Mater. 283-287 (2000) 1385.
[11] B. A. Pint, L. D. Chitwood, and J. R. DiStefano, DOE/ER-0313/35 (2003) 13.
[12] B. A. Pint, K. L. More, H. M. Meyer, and J. R. DiStefano, Fusion Sci. Technol. (in press, 2005).
[13] S. J. Pawel, ORNL/TM-2002/280, Oak Ridge National Laboratory, Oak Ridge, Tenn. (2002).

Table 1. Chemical composition using inductively coupled plasma and combustion analysis of the starting Pb and the Pb-Li after capsule exposures at the indicated temperatures for 1000h (in ppma except for Li in atomic%).[12]

Test	Li	Si	C	O	N	Al	Cr	Fe	Mo	Ni	Y
Starting	n.d.	<40	<170	1270	<40	<8	<4	<4	<2	<4	<2
800°C	17.49%	<30	1850	4090	100	6	<3	6	<2	<3	<2
1100°C	16.27%	<30	1160	3550	90	<6	<3	6	<2	<3	<2