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Journal of Nuclear Materials 307–311 (2002) 1351–1354

journal of nuclear materials

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# Compatibility of materials for fusion reactors with Pb–17Li

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**Abstract**

Tungsten and $SiC_f$/SiC composite are materials considered for fusion reactors. It is therefore required to assess the corrosion behavior of these materials in contact with Pb–17Li. Corrosion tests of tungsten and $SiC_f$/SiC composites in the presence of isothermal stagnant liquid Pb–17Li have been performed. Under the tested conditions, in the 450–800 °C temperature range, the dissolution of tungsten is very limited. Furthermore, no formation of reaction products and no liquid metal penetration have been observed. As far as the $SiC_f$/SiC composite is concerned, no reaction with the liquid has been observed at 800 °C. A Pb–17Li penetration has been detected in the open porosity of the cut material.


## 1. Introduction

Liquid metals are often considered in nuclear applications. However, corrosion by liquid metals is a concern when solids are exposed to this environment. In a fusion reactor, the Pb–17Li eutectic liquid is considered as a liquid coolant due to its high thermal conductivity but also as tritium breeding material of the blanket.

The plasma-facing region is submitted to a high particle flux generated in the plasma and solutions must be found to extract the high heat fluxes to which the high heat flux components, especially the divertor, are submitted. The high thermal conductivity of liquid metals makes the liquid metal-cooled divertors very attractive [1]. Tungsten alloys appear to be the best choice as armor material. As they will be in contact with the coolant, it is required to assess their corrosion behavior in contact with liquid Pb–17Li.

Up to now, the compatibility of tungsten or its alloys with Pb–17Li was not considered in detail. Concerning the solubility of W in Pb–17Li, only a few experiments were performed with W-crucibles. It was concluded that the solubility of this element in the eutectic was less than 1 wppm at 600 °C [2]. Thus, it is thought that tungsten like molybdenum should be practically unattacked in Pb–17Li. However, discrepancies exist about the solubilities determined for some refractory metals in Pb–17Li as in the case of molybdenum, for which Coen et al. [3] reported at 600 °C a 26 wppm solubility value whereas Feuerstein [2] found 0.3 wppm. In addition, during the solid–liquid contact, apart from dissolution, other phenomena such as compound formation at the surface or liquid metal penetration in the solid leading to embrittlement can also occur.

In other respects, the TAURO blanket developed in the European Community by CEA [4] is a self-cooled breeder blanket using the $SiC_f$/SiC ceramic matrix composite as structural material and liquid Pb–17Li as coolant, breeder and neutron multiplier. Among the most significant design issues of the TAURO blanket, the assessment of the compatibility between Pb–17Li and $SiC_f$/SiC (including the brazing material) has been identified. Concerning the compatibility of SiC material with Pb–17Li, a preliminary study has concluded that SiC is unchanged after 1500 h exposure in the alloy at 1073 °C [5]. However, the compatibility of $SiC_f$/SiC composite with molten Pb–17Li has to be investigated more in depth. This paper reports some results on the

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0022-3115/02/$ - see front matter 
PII: S0022-3115(02)00987-X

compatibility of W and $SiC_f$/SiC composite with static isothermal Pb–17Li.

## 2. Experimental procedure

### 2.1. Materials

The tungsten alloy (W–1 wt%$La_2O_3$) was delivered by Plansee company. Its typical composition was W–1 wt%$La_2O_3$ with impurities (wppm). The $SiC_f$/SiC composite was provided by SNECMA Moteurs (SEP division). The three dimensional composite Cerasep® N3-1 was used. It is made of SiC fibers (Nicalon NL207) produced by Nippon Carbon and it is densified by chemical vapor infiltration process (CVI). The chemical composition (wt%) of the SiC fibers is 56.6 Si–31.7 C–11.7 O. The fiber content in the composite is 40% and its porosity is about 10%. For the corrosion tests, one sheet was used as-received and another one was mechanically cut to have naked fibers (in order to detect if this machining alters the corrosion behavior as the composite is not completely densified).

### 2.2. Experimental procedure

After cleaning in acetone and ethanol, weight and geometrical measurements of the specimens were made. Specimens, mounted into a TZM alloy holder were introduced together with about 800 g molten Pb–17Li into a Mo crucible (TZM alloy, inner diameter: 26 mm, height: 200 mm). This operation is carried out in an inert atmosphere glove box. After cooling, the Mo crucible with specimens was sealed by electron beam welding. In order to protect the Mo crucible from oxidation during the test, this latter was placed into nickel alloy or 316 L steel container which was subsequently electron beam welded. After the tests, the container and the crucible were opened, the Pb–17Li melted and the samples extracted from the alloy in the glove box. The weight variations of the corrosion samples after the tests have not been measured because the tungsten and composite materials were attacked by the chemical mixture (1/3 ethanol, 1/3 acetic acid, 1/3 hydrogen peroxide) used to remove the lead alloy adhering to the specimen surface. The surface and the cross section of the specimens after exposure to Pb–17Li were observed by SEM.

After the tests, samples of frozen Pb–17Li were analysed by atomic absorption.

### 2.3. Experimental conditions

The tungsten corrosion tests were performed at 450, 600 and 800 °C in isothermal conditions for 1500 h. The $SiC_f$/SiC composite corrosion test was performed at 800 °C for 3000 h in isothermal conditions.

## 3. Results

### 3.1. Corrosion of tungsten

The interaction of a solid with a liquid can be characterized by the analysis of the melt after the corrosion tests. The Mo concentrations in Pb–17Li after all the tests are between 2 and 3 wppm. These analyses indicate that the Mo crucible is not completely inert towards the Pb–17Li alloy.

The tungsten concentration in Pb–17Li at the end of the three tests is very low (<0.5 wppm W) and no change is detected with the test temperature. Such values might indicate that the dissolution of tungsten in Pb–17Li is not important. However, we must draw attention to the experimental conditions. Under isothermal conditions, the element concentration in the liquid becomes equal to the solubility after a period of time. Assuming the solubility of W in Pb–17Li is very low, the dissolution rate can decrease with time and reach zero. Therefore, the corrosion of tungsten by the direct dissolution process can be minimized in our tests and corrosion experiments in non-isothermal conditions would be necessary.

Apart from dissolution effects, the changes in surface morphology and composition due to liquid metal penetration or compound formation are important to consider. Cross sections of the samples were thus observed by SEM. It has to be noted that all the SEM observations were made without removing the lead alloy adhering to the surface (i.e., there was no cleaning with the chemical mixture before the analysis). The observation of the surface of tungsten before exposure to the melt is shown in Fig. 1 as a reference. Fig. 2 shows the solid–liquid interface after exposure at 800 °C: no attack is detected. For all the tested temperatures, no reaction products have been observed at the interface by X-ray analysis. These results are different of those obtained

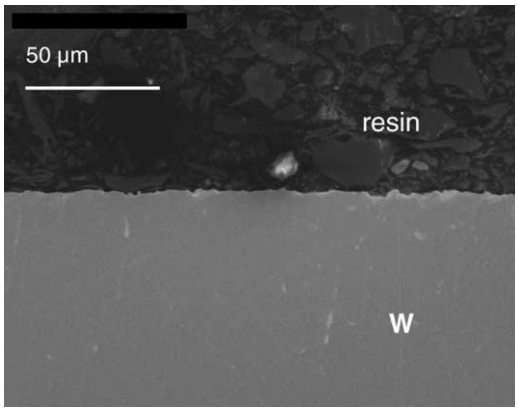


Fig. 1. Surface of the W alloy before exposure to the Pb–17Li liquid alloy.

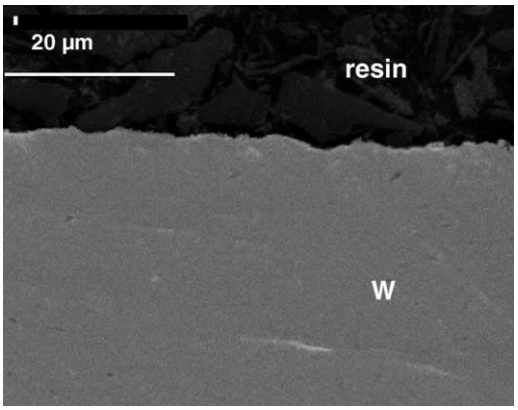


Fig. 2. Cross section of the W alloy after 1500 h exposure at 800 °C in Pb–17Li.

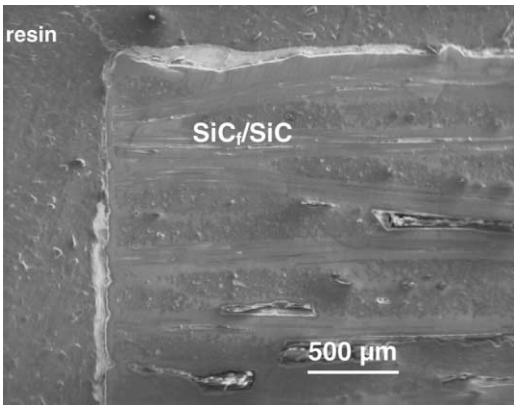


Fig. 3. Cross section of the as-received $SiC_f$/SiC specimen after 3000 h exposure at 800 °C to the Pb–17Li liquid alloy.

with tungsten in contact with pure liquid lead [6]. In that case, it was found that the presence of oxygen in lead modifies the interaction mechanism leading to the oxidation of tungsten. Tungsten oxide is in fact more stable than lead oxide. In the case of the Pb–17Li alloy, the solubility of oxygen is very small [7] and the oxygen concentration is much lower than that contained in the molten lead used in [6]. Moreover, a fundamental characteristic is the presence of lithium in the alloy: the oxygen affinity for lithium is higher than for tungsten so that lithium oxide is more stable than tungsten oxide. As a consequence, tungsten cannot be oxidized in contact with Pb–17Li and our results agree well with that.

Another concern when solids are exposed to molten metals is the penetration of the liquid along the grain boundaries. The formation of a liquid channel from the surface of tungsten has not been observed neither on the cross section nor on the X-ray images. Nevertheless, further investigations about the microstructure of tungsten are needed to definitely conclude about Pb–17Li penetration along the W grain boundaries.

### 3.2. Corrosion of $SiC_f$/SiC composite

After the corrosion test at 800 °C, the Pb–17Li alloy was analysed. Preliminary analyses indicated about $(8 \pm 4)$ wppm for Si in Pb–17Li. Concerning the solubility of silicon in liquid lead, data are very limited and contradictory. Molten Pb could dissolve small quantities of Si at high temperatures (200 wppm of Si in Pb at 1250 °C [8]) but it seems that up to 900 °C, it is not much soluble.

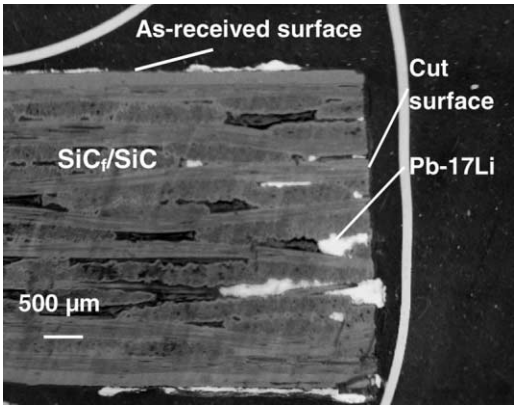


Fig. 4. Cross section of the $SiC_f$/SiC specimen after exposure to the Pb–17Li liquid alloy. The vertical side of the section corresponds to the cut surface. The horizontal side corresponds to the as-received surface.

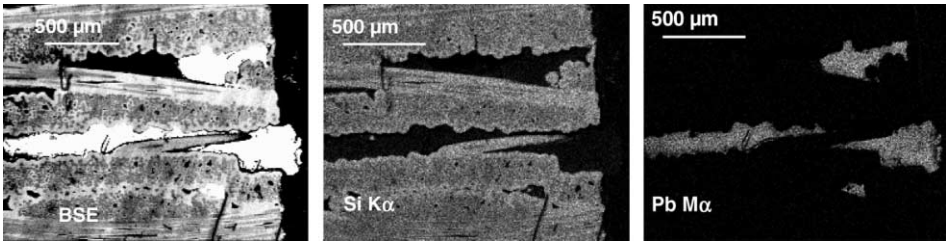


Fig. 5. X-ray maps of the cross sectioned $SiC_f$/SiC specimen (cut surface exposed to Pb–17Li).

The specimens exposed to Pb–17Li were examined by SEM without removing the lead alloy adhering to the surface.

Concerning the surfaces which were not modified (as-received surfaces), the cross section presented in Fig. 3 does not show significant open porosities from the two sides of the specimen. The X-ray maps do not exhibit liquid penetration into the composite: the lead alloy is only adherent to the surface. Therefore, no corrosion damage is observed for the $SiC_f$/SiC material exposed to Pb–17Li.

The cross section of the surface which was cut after manufacturing (cut surface) shows that penetration of Pb–17Li has occurred into the open porosities (Fig. 4). The presence of the liquid alloy in the free space is confirmed on the different X-ray maps (Fig. 5).

## 4. Conclusion

The corrosion behavior of a tungsten alloy has been studied by isothermal immersion tests in static liquid Pb–17Li in the temperature range 450–800 °C and for exposure times up to 1500 h. The amount of tungsten found in Pb–17Li after all the corrosion tests was very small (<0.5 wppm) indicating that the dissolution of this element is likely very limited. However, it has to be noted that the dissolution process may be minimized in the present isothermal tests due to the very low solubility of tungsten in the melt. In order to evaluate the dissolution rate, tests should be carried out in non-isothermal conditions. The interaction with the molten alloy did not lead to the formation of reaction products at the tungsten surface. No attack and no composition change were found at the solid–liquid interface. The preliminary observations did not exhibit any liquid metal penetration in the tungsten. Nevertheless, the microstructural study must be carried out more in depth. Further investigations are required for a complete evaluation of the corrosion behavior by using more sophisticated analytical equipment.

The preliminary experiments with the $SiC_f$/SiC composite Cerasep® N3-1 exposed to static isothermal liquid Pb–17Li at 800 °C for 3000 h indicate that this material does not react with the liquid alloy. Therefore, $SiC_f$/SiC composite should be stable and compatible with this environment at this temperature. A penetration of Pb–17Li was only observed into the free space (porosity) as the composite is not highly densified. The open porosity was mainly due to the cutting of the specimen after manufacturing. A more complete study is necessary to investigate the effect of the machining on the liquid infiltration into the composite. Moreover, the effects of the temperature and liquid flow velocity on the corrosion behavior of this material must be evaluated.

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