Journal of Nuclear Materials 392 (2009) 259–263

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

An X-ray absorption spectroscopy study of Mo oxidation in Pb at elevated temperatures

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A B S T R A C T

The corrosion of fuel cladding and structural materials by lead and lead-bismuth eutectic in the liquid state at elevated temperatures is an issue that must be considered when designing advanced nuclear systems and high-power spallation neutron targets. In this work, lead corrosion studies of molybdenum were performed to investigate the interaction layer as a function of temperature by X-ray absorption spectroscopy. *In situ* X-ray absorption measurements on a Mo substrate with a 3–6 μm layer of Pb deposited by thermal evaporation were performed at temperatures up to 900 °C and at a 15° angle to the incident X-rays. The changes in the local atomic structure of the corrosion layer are visible in the difference extended X-ray absorption fine structure and the linear combination fitting of the X-ray absorption near-edge structure to as-deposited molybdenum sample and molybdenum oxide (MoO_2 and MoO_3) standards. The data are consistent with the appearance of MoO_3 in an intermediate temperature range (650–800 °C) and the more stable MoO_2 phase dominating at high and low temperatures.

1. Introduction

Lead and lead-bismuth eutectic (Pb/LBE) have emerged as primary candidates for application in advanced nuclear reactors and transmutation systems as coolants and high-power spallation neutron targets [1–5]. Pb/LBE has a low melting temperature, high boiling temperature and it is chemically inert. However, Pb/LBE is very corrosive to many structural materials, if they are exposed to Pb/LBE directly at medium to high temperatures. This has limited the useful operating range of most Pb/LBE eutectic systems to temperatures around 550–600 °C.

Studies of the corrosion reactions in the Pb/LBE system are important in developing mitigation techniques, corrosion tolerant materials, and coatings. It is known that the formation of a protective oxide layer on the metal surface using controlled amounts of oxygen can protect against dissolution attack by liquid lead-alloys [1]. Liquid metal corrosion of steels has been shown [6–13] to be dependent upon many different parameters including the type of steels, thermal conditioning, the surface treatment of steel, oxygen concentration, application temperature, flow velocity, etc. Fazio et al. [14] and Ilincev et al. [15] reported on the corrosion of steel and refractory metals (W, Mo) by oxygen-controlled flowing LBE. Mo and W, both refractory metals, have the lowest solubility in LBE and exhibit better corrosion resistance than steels. Since corrosion occurs at the surface, corrosion resistance has been increased by alloying Al, Si, or Zr into the steel surface or by maintaining protective films formed by carefully controlling the dissolved oxygen concentration in the molten Pb/LBE [16–22]. However, definite and quantitative methods for preventing corrosion cannot be presented because experimental results are either scarce or when available are under varying conditions.

In this paper, we report the characterization of the local atomic and electronic structures of the corrosion products with molybdenum by X-ray absorption spectroscopy (XAS). XAS is the modulation of the X-ray absorption probability of an atom due to the chemical and physical state of the atom. The XAS is typically divided into two regimes: X-ray absorption near-edge spectroscopy (XANES) and extended X-ray absorption fine-structure spectroscopy (EXAFS). XANES provides qualitative information about formal oxidation state and coordination chemistry of the absorbing atom, while EXAFS allows the determination of the distances, coordination number, and types of near neighbor atoms. XAS has rarely been used to study the properties of Pb corrosion tolerant materials and coatings in the past. However, we show that it is possible to determine the structural changes at the Pb–Mo interface after high temperature treatment. Such an atomistic understanding of the corrosion process can be extended to other systems and will assist in improving the development of corrosion resistant materials and coatings.

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doi:10.1016/j.jnucmat.2009.03.005