§11. Control of Nitrogen Concentration in Liquid Lithium by Iron-Titanium Alloy

Tanaka, S., Hirakane, S., Yoneoka, T. (Univ. of Tokyo), Muroga, T.

Reducing the nitrogen concentration in liquid lithium is one of the most important steps in creating a liquid lithium blanket system. In this study, in order to verify the nitrogen gettering performance of Fe-Ti alloy, the variation in the nitrogen concentration in liquid lithium, into which Fe-10at.%Ti or Fe-5at.%Ti getter was immersed, was examined. In the experiments, the alloys were cut into plates of 1 x 10 x 40 mm³. The mass of the lithium was 25 g. The initial nitrogen concentration in liquid lithium was adjusted to 90-280 wtppm by contacting N₂ gas with the liquid lithium (Sieverts' method). The experiment was conducted by using a molybdenum crucible in a stainless-steel pot in the glove box filled with argon gas. The nitrogen concentration in the sampled lithium was measured using the ammonia method.

Figure 1 shows the variation in the nitrogen concentration in liquid lithium at 873 K under different immersion conditions. Based on the results, it was concluded that Fe-Ti alloy absorbs the nitrogen in the liquid lithium, although the effect is not durable, and that Fe-5at.%Ti possesses a better gettering performance than Fe-10at.%Ti. The total quantities of absorbed nitrogen for initial nitrogen concentration of 280 wtppm are comparable to the amount of titanium existing between the surface and a depth of several μm , provided that the absorbed nitrogen is trapped by titanium as TiN. Based on the fact that the nitrogen concentration in liquid lithium decreased from the initial 100 wtppm to 30 wtppm due to both Fe-10at.%Ti and Fe-5at.%Ti, Fe-Ti alloy was determined to be effective even when the nitrogen concentration is low. Note that the value of 30 wtppm is almost equal to the measurement limit. In order to clarify the effect of temperature on the gettering performance, the nitrogen concentration in liquid lithium with the Fe-Ti alloys at 723 K was measured until the variation stopped. Then, the temperature was changed to 873 K and the concentration was measured again until the variation stopped. The result of this experiment is given in Fig. 2. The nitrogen concentration at which the variation stopped at 873 K was lower than that at 723 K in both Fe-Ti alloys. This temperature dependence indicated that some absorbed nitrogen can migrate easily between the liquid lithium and the Fe-Ti alloy.

XRD spectra were obtained of the Fe-Ti alloys before and after the immersion test at 873 K with the initial nitrogen concentration of 250 wtppm. Before the immersion, $\alpha\text{-Fe}$ and Fe₂Ti peaks were detected for Fe-10at.%Ti, while only $\alpha\text{-Fe}$ peaks were observed for Fe-5at.%Ti. After immersion, TiN peaks emerged for both Fe-10at.%Ti and Fe-5at.%Ti. In addition, a decrease in the Fe₂Ti peaks was noticed for Fe-10at.%Ti. These

observation suggest that some of the absorbed nitrogen formed TiN by consuming the titanium in the Fe₂Ti of Fe-10at.%Ti, and by combining with the titanium dissolved in the $\alpha\text{-Fe}$ matrix of Fe-5at.%Ti.

XMA images of the sample were also studied. For Fe-10at.%Ti sample after the immersion test, a zone considered to be TiN was detected only near the surface, at a depth less than several μm . The distribution of titanium on the surface was determined by XMA before and after the immersion. The localization of titanium was also determined. This localization could have been caused by the gathering of the TiN generated by the nitriding of Fe₂Ti in the surface region. Since some regions of the surface did not contain TiN, it is reasonable to assume that something other than TiN coverage of the surface impeded the nitrogen absorption.

XPS spectra were taken after the immersion test during the surface sputtering for Fe-10at.%Ti. TiN peak at 396.7eV was observed at a depth of 200-4000nm. The concentration of the TiN peak was smaller at 0-200nm than that at 200-4000nm.

Temperature dependence of the attained nitrogen concentration (Fig. 2) and the limit of nitrogen concentration in the case of initial nitrogen concentration of 280 wtppm were discussed from these results and an internal diffusion barrier was thought to exist in the bulk of the alloy to impede nitrogen diffusion.

Fig. 1 Variations in the nitrogen concentration in liquid lithium at 873K.

Fig. 2 Temperature dependence of the nitrogen concentration in liquid lithium.