§ 25. The Control of Nitrogen Impurity in Lithium by Hot Trapping

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Impurities in IFMIF liquid lithium include accumulated nuclear reaction products of the facility operation and impurities from the cover gas and structural materials. Among these impurities, nitrogen and hydrogen are difficult to be removed by cold trapping methods. If yttrium is used as a getter material for tritium, nitriding of the yttrium surface can prevent tritium being absorbed.

One of the purposes of the present study is to control the nitrogen concentration down to 10^{-3}mass% in liquid lithium by the method of hot trapping. Due to the difficulty of accurate nitrogen analysis under 5×10^{-3}mass% in lithium, we are forced to evaluate the possibility of gettering under this low nitrogen concentration from the experimental data that initial nitrogen concentration in molten lithium are from 10^{-2}mass% to 10^{-1}mass%. Nitriding of yttrium surface is considered to prevent tritium being absorbed. Therefore, one purpose of the present study is to investigate the behavior of nitrogen in the system of V-Ti alloy and yttrium in liquid lithium.

Vanadium, Titanium, three types of alloy (V-5at%Ti, V-10at%Ti and V-15at%Ti), and chromium were used for the getter materials. The surface area of these gettering metals used for each run was from 20cm^2 to 25cm^2. The mass of lithium was mainly 25g. The purity of it was higher than 99.9%. The concentration of nitrogen impurity in the as-received lithium was mainly 5×10^{-3}mass%. In most of experiments, the nitrogen concentrations in the lithium were increased to about 3.7×10^{-2}mass% by the Sievert's method. The experimental temperature was from 673K to 873K and the immersion periods were 0.26Ms (3days) to 2.8Ms (32days). Change of the nitrogen concentration in the liquid lithium by gettering was measured by the ammonia method. The getter materials and yttrium specimen were also immersed into the liquid lithium. The experimental temperature was 823K and the duration was 0.5 Ms.

For the experimental results of nitrogen gettering up to 823K, the V-10at%Ti specimen showed most efficient material among the vanadium, titanium and these alloys except V-15at%Ti. But, at 823K, the nitrogen gettering rate of chromium was found to be larger than that of the V-10at%Ti alloy. In addition, these reducing rates of nitrogen in the molten lithium seem to be somewhat slow. For increasing the reducing rate, the tests at 873K were carried out. The specimens were V-10at%Ti alloy, chromium, and V-15at%Ti alloy. The two V-Ti alloys reduced nitrogen from about 3.7×10^{-2}mass% of the initial concentration in lithium to below the half values of the initial concentration in initial 86.4ks (1day). On the other hand, chromium reduced nitrogen to 60% of the initial concentration in initial 86.4ks. In any case, these decreasing rates were much larger than that at 823K. Finally, the gettering parabolic rate constant for V-10at%Ti alloy at 873K was obtained as $2\mathrm{g/m^2}\cdot(\mathrm{day})^{1/2}$. From the typical XRD analysis, a VN-TiN (nitride of titanium and vanadium) layer was formed on the surface of the V-10at%Ti alloy that was immersed at 823K for 2.8Ms. In addition, widely broad peaks of the V-Ti-N solid solution were also confirmed. When the surface of this alloy was polished by 4µm in depth, peaks of the VN-TiN layer disappeared and only those of the nitrogen solid solution was confirmed. When the surface was polished by 8µm, shifted and broad peaks almost disappeared and the XRD peaks backed to the sharp one like as received specimen. From these facts, the gettering effect seemed to be mainly caused by the solution on nitrogen in alloy.

The long-term change of the nitrogen concentration in the liquid lithium by chromium gettering was measured by the ammonia method. The nitrogen gettering rate by the chromium specimen becomes slower below 7×10^{-3}mass% of the nitrogen concentration, and it seemed to approach a limit of about 6.5×10^{-3}mass%. The reason for this limit is believed to be the instability of the ternary nitride of chromium and lithium below this nitrogen concentration.

The immersion experiments of yttrium plates in lithium without and together with V-10at%Ti alloy were performed. The experimental conditions were 823k in temperature and 0.5Ms in time. After the immersion, the yttrium nitride was found on the surface of the specimen by XRD measurement. Peaks of the yttrium nitride immersed with the V-10at%Ti alloy become smaller, compared with the result immersed without the V-Ti alloy.

In conclusion,

1) Under low nitrogen concentration in liquid lithium, V-Ti alloy has large nitrogen gettering effect at 823K and 873K
2) Gettered nitrogen by the V-Ti alloy formed mainly the solid solution with this alloy
3) Under high nitrogen concentration in liquid lithium, nitrogen gettering by chromium is effective compared with V-Ti alloy
4) Nitrogen gettering by chromium at about 850K reached a limit at 6.5×10^{-3}mass% of nitrogen concentration in liquid lithium
5) Nitriding of yttrium in liquid lithium was suppressed by immersion together with V-10at%Ti alloy