§5. Control of Nitrogen Impurity in Liquid Lithium by Hot Trapping

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Nitrogen concentration in liquid lithium was controlled by the method of hot trapping. In the year 2000, Ti and V-Ti alloy were tested and V-10at%Ti was found to be most effective. In the year 2001, trapping velocity by V-10at%Ti was tested by measuring the change of nitrogen concentration in liquid lithium. Chromium was also tested, because it was expected to be effective at high concentration of nitrogen by making nitride.

V-10at%Ti was a plate with the size of 1x10x40mm. Purity of Cr was higher than 99.9%, and rod (1.5x4x30mm) was used. Nitrogen concentration in the prepared lithium was 50 wppm. In some experiments, nitrogen was added to liquid lithium by the Sieverts’ method. All experiments were conducted in the glove box covered with Ar gas. After immersion in liquid lithium, gettering materials in the crucible were recovered and then lithium adhered to the surface was removed by washing with water or methyl alcohol. Mass measurement, XRD analysis and nitrogen concentration detection were conducted for the gettering materials. Nitrogen concentration in liquid lithium was measured by an ammonium method.

Figure 1 shows the increase of nitrogen concentration in gettering materials. Initial nitrogen concentration in liquid lithium was 100wppm for V and Ti, and 50wppm for V-Ti alloys. This figure shows that V-10at%Ti was most effective among tested. From XRD analysis, with the surface being etched, it was concluded that nitrogen exists as thin nitride near the surface and as solid solution with high solubility limit in the bulk.

From the nitrogen concentration change for V-10at%Ti, nitrogen diffusion in the getter was considered as the controlling process. This means that V-10at%Ti can get nitrogen to a lower level, several ppms. From the decreasing rate of nitrogen concentration in liquid lithium combined with the smaller surface area of the Cr specimen, it was concluded that gettering by chromium is more effective than by V-10at%Ti in the nitrogen concentration range of several 100 wppm.

![Figure 1. Nitrogen concentration increase](image1)

Fig. 1. Nitrogen concentration increase

Figure 2 shows the change of nitrogen concentration in liquid lithium during hot trapping by V-10at%Ti and Cr.

![Figure 2. Change of nitrogen concentration in liquid lithium during hot trapping by V-10at%Ti and Cr](image2)

**Fig. 2. Change of nitrogen concentration in liquid lithium during hot trapping by V-10at%Ti and Cr**

Figure 3 indicates the existence of the lower limit (ca. 65wppm) of nitrogen concentration in liquid lithium when Cr was used as a gettering material, because nitride of Cr is not stable at the nitrogen concentration below this level. The composition of this nitride was suggested to be Li₆Cr(III)₃N₅ from the ICP and ion chromatography analyses for the solution to which the surface layer formed on the Cr getter was dissolved.

![Figure 3. Change of nitrogen concentration in liquid lithium during hot trapping by Cr](image3)

**Fig.3. Change of nitrogen concentration in liquid lithium during hot trapping by Cr**

Reference