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マイクロ波水素プラズマを用いて水素化されたMg酸化物表面の解析
Analysis of Magnesium Oxide Surfaces Hydrogenated by H_2 Microwave Plasma

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1. Introduction

Sodium borohydride ($NaBH_4$) is attracting attention for a regeneratable and large capacity powdered hydrogen carrier. $NaBH_4$ generates sodium metaborate ($NaBO_2$) as a by-product of dehydrogenation. Here, Magnesium hydride (MgH_2) is important for a regeneration from $NaBO_2$ to $NaBH_4$ with ball milling process and becomes Mg oxides after the reaction[1]. We confirmed that Mg oxides was hydrogenated by using low-temperature microwave excited hydrogen plasma. In the plasma processing, a hydrogen radical (H^*) and ion (H^+) will contribute to the hydrogenation of Mg oxides. However, the dehydrogenation from the hydrogenated Mg surface also occurs simultaneously. The dehydrogenation is much dependent on the surface temperature of the hydrogenated Mg. Therefore, the effect of the hydrogen ion dose and sample temperature *T* on the surface region of hydrogenated Mg by plasma was investigated by Raman spectroscopy, X-ray photoelectron spectroscopy (XPS).

2. Experimental Methods

Naturally oxidized Mg plates were treated by 2.45 GHz microwave excited hydrogen plasma. The microwave power and gas pressure were kept constant at 500 W and 9-15 Pa, respectively. *T* was measured with a thermocouple installed on a back surface of a MgO pellet. The ion dose was calculated by the product of treatment time *t* and ion flux Γ_i at the treatment sample position *z*. Γ_i was measured with a Langmuir probe. The surface region on hydrogenated Mg was determined by results of 2D Raman mapping spectroscopy analysis.

3. Results and Discussion

Figure 1 shows the condition map of plasma treated samples in *z* and *t* axis with isolines of *T* and the ion dose. As a result of hydrogen plasma treatment, MgH_2 appeared at the ion dose of $1.0\text{-}3.0\times10^{19}$ cm^{-2} and *T* of 150-200 ℃. When the ion dose and *T* were further increased, MgH_2 disappeared and then $Mg(OH)_2$ appeared. It is speculated that dehydrogenation of MgH_2 increases with *T* by the ion dose, and $Mg(OH)_2$ appeared by reacting with oxides generated by the reduction of Mg oxides. Figure 2 shows the Raman mapping image of synthesized MgH_2 at the treatment condition of *z*=80 mm and *t*=20 min, which condition has the largest synthesized MgH_2 region in the conditions under which MgH_2 was synthesized in Fig. 1. It is speculated that the growth of MgH_2 requires hydrogen ion dose and suppression of *T* which prevent dehydrogenation.

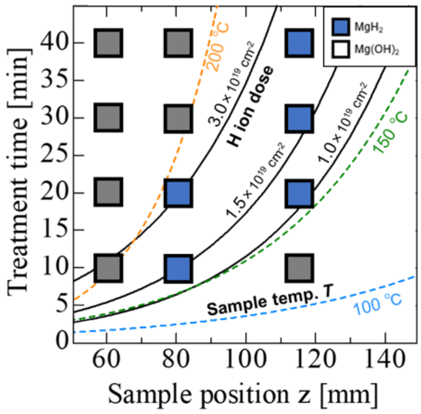

Fig. 1. Condition map of plasma treated samples with *T* and the ion dose.

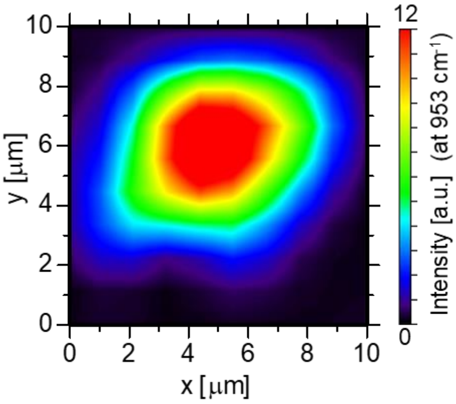

Fig. 2. The Raman mapping image of the surface region of synthesized MgH_2 by H_2 plasma.

References

[1] C. Hsueh, et al., Int. J. Hydrog. Energy **34** (2009) 1717.