

## Study on the effects of hydrogen decrepitation on the formation of $\text{Sm}_2\text{Fe}_{17}\text{N}_x$ -type material

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### $\text{Sm}_2\text{Fe}_{17}\text{N}_x$ -type 재료의 형성에 미치는 수소파쇄의 영향에 관한 연구

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#### 1. Introduction

The  $\text{Sm}_2\text{Fe}_{17}\text{N}_x$ -type material has overall magnetic properties comparable to those of the Nd-Fe-B-type counterpart. In the period since the discovery of the nitride material a variety of research efforts have been made to improve the magnetic properties and to develop an effective production routes. More recently, the Sumitomo Metal Mining, Japan, has announced that they introduced the injection moulded bonded anisotropic  $\text{Sm}_2\text{Fe}_{17}\text{N}_x$ -type magnet with energy product of 13 MGOe into market. It is generally understood that one of the difficulties encountered in producing the nitride material is the poor kinetics of the nitride formation reaction. The  $\text{Sm}_2\text{Fe}_{17}$  alloy is, therefore, processed into a powder-like material in order to improve the reaction kinetics, and it was reported that the hydrogen decrepitation (HD hereafter) treatment was an effective way of producing fine powder of the  $\text{Sm}_2\text{Fe}_{17}$  alloy. In the present study, in an attempt to find an effective production way of the  $\text{Sm}_2\text{Fe}_{17}\text{N}_x$ -type material the  $\text{Sm}_2\text{Fe}_{17}$ -type alloy was subjected to a HD treatment prior to a nitrogenation, and its effect on the formation of the nitride material has been investigated.

#### 2. Experimental Work

The alloy used in the present study has a chemical composition of Sm 22.7 wt.%, Fe 72.3 wt.%, Nb 5.0 wt.%. The as-cast alloy was subjected to a hydrogen decrepitation prior to nitrogenation. The alloy was hydrogenated (hydrogen pressure: 1.5 bar) at 300 °C for 1 hour and then degassed at 450 °C for 4 hours under vacuum, and these treatments were repeated four times to cause a severe decrepitation. The HD-treated alloy was pulverised for 1 hour. The as-cast alloy was also pulverised under the same condition for comparison. The obtained powder materials were then nitrogenated at 475 °C under nitrogen gas (nitrogen pressure : ~1 bar). The nitrogenation behaviours of the alloy were investigated using a TPA (thermopiezic analysis), TMA, XRD and DTA under nitrogen gas. The magnetic properties of the nitride material were characterised by means of VSM.

### 3. Results and Discussion

Fig. 1 shows the TMA results for the materials nitrogenated at 475 °C for 4 hours using an as-cast or HD treated alloy powders. It appears that for the material produced from the as-cast alloy two deflections are observed at around 150 °C and around 470 °C, respectively. Those reflections may be corresponding to the Curie temperatures of the unreacted  $\text{Sm}_2\text{Fe}_{17}$  phase and the formed  $\text{Sm}_2\text{Fe}_{17}\text{N}_x$ -type nitride, respectively. Meanwhile, for the materials produced using the HD-treated alloy only one deflection at around 470 °C appears, indicating that the alloy has been fully nitrogenated under the condition used. These results indicate clearly that the previous HD treatment may facilitate the formation of  $\text{Sm}_2\text{Fe}_{17}\text{N}_x$ -type nitride. Fig. 2 shows the XRD spectra for the materials nitrogenated under identical condition (475 °C for 4 hours) using the as-cast or HD-treated alloy. Also included in the Fig. 2 is the spectrum for the as-cast alloy for comparison. It can be seen that the nitride produced from the HD-treated material has diffraction peaks moved consistently toward lower angle with respect to the as-cast alloy. This indicates that the HD-treated alloy has been fully nitrogenated under the condition used. Meanwhile, the material nitrogenated using the as-cast alloy shows split diffraction peaks. This indicates that the material has been partially nitrogenated, and the unreacted  $\text{Sm}_2\text{Fe}_{17}$ -type and formed  $\text{Sm}_2\text{Fe}_{17}\text{N}_x$ -type phases coexist. These results also confirm that the previous HD treatment may facilitate the formation of  $\text{Sm}_2\text{Fe}_{17}\text{N}_x$ -type material. It has also been found that the hydrogen atoms existing in the initial HD-treated alloy were removed almost completely during the nitrogenation. The heat output associated with the nitrogenation of the previously HD-treated alloy was found to be significantly smaller with respect to the as-cast alloy.

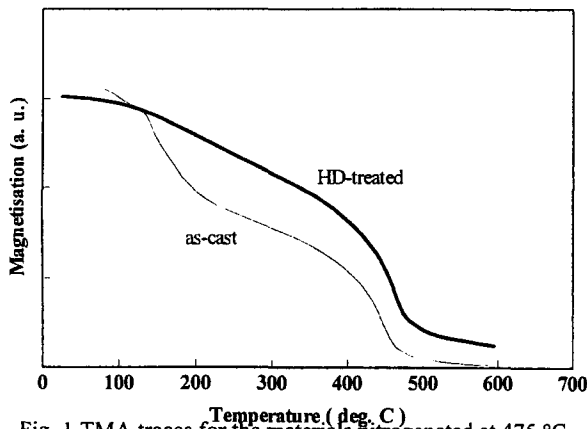


Fig. 1 TMA traces for the materials nitrogenated at 475 °C for 4 hours.

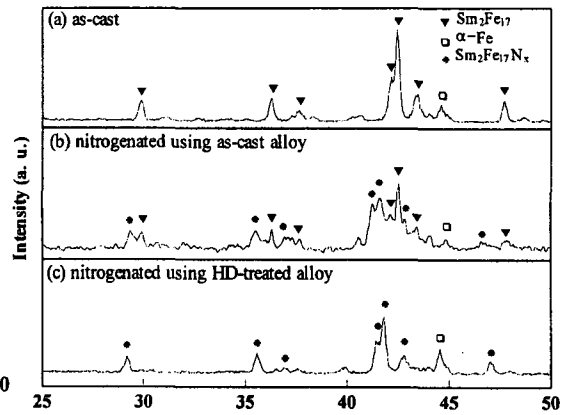


Fig. 2 XRD spectra for the materials nitrogenated at 475 °C for 4 hours.