論　文　要　旨
Thesis Abstract

(yyyy/mm/dd)　2021 年 2 月 15 日

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主論文題名　（Title）

Morphology-Controlled Synthesis of Nitrides from Titanium Dioxide and Titanium Disilicide by Ammonia Nitridation

内容の要旨　（Abstract）

The objective of this thesis is to investigate the methods of preparing TiN, one of the essential refractory materials and industrially used, to obtain the product with less agglomerated and defined shape particles. Additionally, the optimum conditions for preparing the TiN-Si_3N_4 composite powders were also investigated in an atmosphere of NH_3.

This thesis consists of five chapters.

Chapter 1 is the general introduction, which contains;

- Information about the nitride materials, their structure, properties, applications, and their famous preparation methods.
- Integrated information about TiN and Si_3N_4 that explain the characteristics of these materials and their industrial importance in various fields.
- General information about PTFE and $TiSi_2$.

In Chapter 2, the effect of the exposure time of the nanocrystalline TiO_2 to NH_3 gas during the nitridation process on the degree of agglomeration of the resulting nanocrystalline TiN was investigated. Nanocrystalline TiN was prepared via direct ammonia nitridation of nanocrystalline TiO_2 powder at 900 and 1,000 °C. The weight changes caused by this nitridation

were monitored by TG. The TG results showed two-step weight losses. Based on the XRD and XPS results, the first and second weight losses were from 750 to 950 °C and beyond 950 °C, respectively. These corresponded to the transformations from TiO_2 to TiO and/or TiO_xN_y to TiN, respectively. During heating in NH_3, oxygen was eliminated, which inhibited sintering of the TiO_2 particles. This sintering inhibition resulted in the formation of less agglomerated nano-sized TiN particles from nano-sized TiO_2.

In Chapter 3, a simple preparation method for simultaneously synthesized TiN–Si_3N_4 mixed materials was obtained by the direct nitridation of $TiSi_2$ under an NH_3 gas flow. Using this nitridation, a mixture of TiN particles and Si_3N_4 fibers were obtained, and, we achieved a 50.9% weight gain at 1,300 °C for 10 h. The nitridation was almost completed, but other reactions, such as oxidation owing to the presence of SiO_2 have occurred. The amount of SiO_2 decreased alongside an increase in holding time. This high reaction ratio was presumed to have been the result of the presence of hydrogen, formed by the thermal dissociation of NH_3.

In Chapter 4, the synthesis method of TiN nanoparticles by the direct nitridation process of $TiSi_2$ under an NH_3 gas flow with the addition of PTFE vapor was investigated. The experimental results showed that the Si formed on the $TiSi_2$ reacts with C_2F_2 (g), which is formed from PTFE decomposition, and form SiF_4 (g), and the Ti in $TiSi_2$ is reacted with C_2F_2 (g) to form a volatile TiF_4, which react with NH_3 to form nanocrystalline TiN.

Chapter 5 is a summary of this thesis.

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