Chemical properties of thorium nitrate hydrate: XPS, Raman, and SEM studies

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1. Introduction

Thorium has attracted attention as a potential fuel material because it is a common element in nature and generates a small fraction of minor actinides compared with uranium during operation [1-2].

Studies on ThO_2 and ThO_2-UO_2 were performed to evaluate the properties of thorium by several groups [1-5]. However, the characteristic of thorium nitrate hydrate ($Th(NO_3)_4$) has been barely investigated in spite of the material being composed of thorium. It is worth being studied to reveal its properties because of the accumulation of fundamental knowledge regarding the thorium element.

Herein, we present the results obtained from using X-ray photoelectron spectroscopy (XPS), Raman spectroscopy, and scanning electron microscopy (SEM), which provide information on the chemical properties of $Th(NO_3)_4$.

2. Experimental Details

$Th(NO_3)_4$ pellets (ϕ : 6.35 mm) were manufactured by thorium nitrate hydrate powder.

XPS result was obtained in a high-vacuum chamber equipped with a VG Scientific ESCALAB 220i-XL system using an Al Kα X-ray source (1486.6 eV) at a base pressure below 6.0×10^{-8} torr. The XPS spectrum was recorded with a pass energy of 100 eV and an energy step of 0.5 eV. The binding energy of the spectrum was calibrated relative to that of adventitious C 1*s* (284.6 eV).

The Raman spectrum was measured by a SR303i Raman spectrometer with a 632.8 nm excitation wavelength helium-neon laser operating at 8 mW. The spectrum was acquired using an exposure of 300 s over a wavenumber range of 190 to 1000 cm^{-1}. The laser was focused onto a sample using 50× uncoated-objective lenses.

An SEM experiment was performed without any sample treatment using a JEOL JSM-6610LV.

3. Results

To research the electronic structure of $Th(NO_3)_4$, we carried out an XPS experiment. Figure 1 presents the XPS survey spectrum obtained from a $Th(NO_3)_4$ pellet at room temperature. All of the peaks in Figure 1 are clearly assigned as Th 4$f_{7/2}$, Th 4$f_{5/2}$, Th 5$d_{5/2}$, Th 5$d_{3/2}$, Th 4$d_{5/2}$, Th 4$d_{3/2}$, N 1*s*, and O 1*s* with adventitious carbon (C 1*s*).

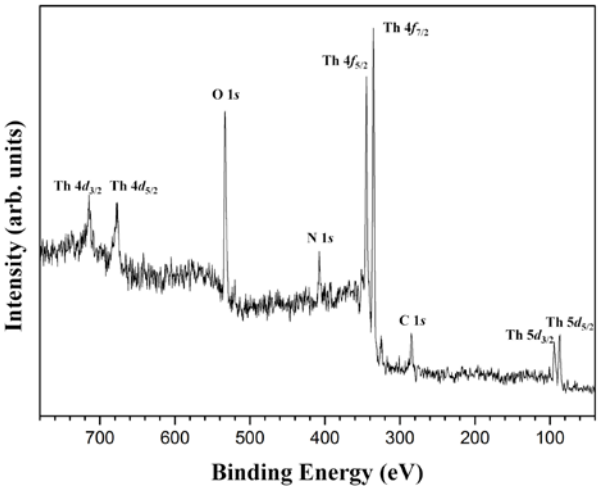

Fig. 1. XPS survey spectrum obtained from a $Th(NO_3)_4$ sample, in which thorium, nitrogen, and oxygen peaks appear with adventitious carbon signal.

Raman spectrum of $Th(NO_3)_4$ pellet at room temperature is described in Figure 2. Previous studies revealed that peaks related to thorium-oxygen stretching vibration, vibration in anhydrous $Th(NO_3)_4$, complexed nitrate, and free nitrate appear at 221, 244, 718, and 753 cm^{-1} in a Raman spectrum, respectively, which correspond to our result [6-7].

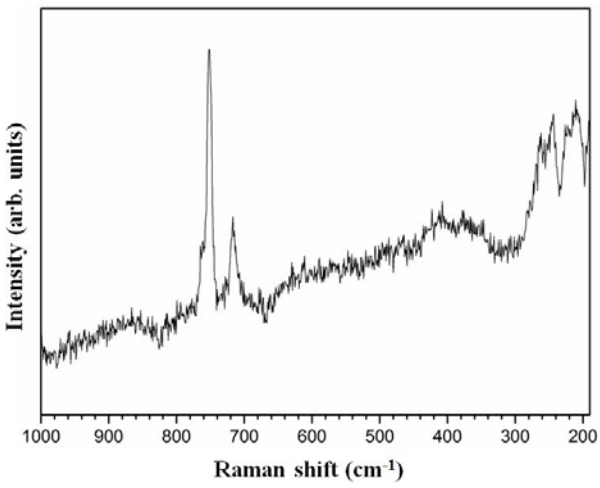

Fig. 2. Raman spectrum of $Th(NO_3)_4$ pellet obtained at room

temperature.

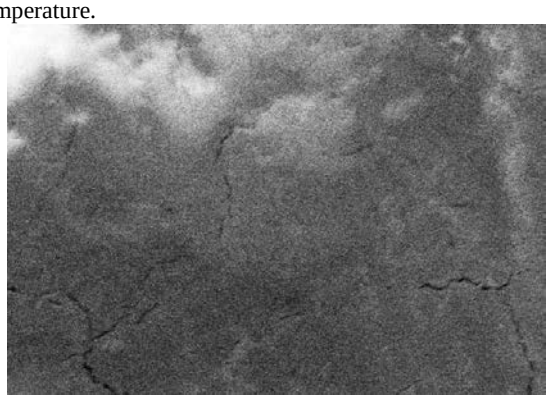

Fig. 3. SEM image (×850) of $Th(NO_3)_4$ pellet at room temperature. Bright areas are caused by a charging effect because the $Th(NO_3)_4$ sample is an insulator.

Finally, we executed an SEM experiment at room temperature. Because the $Th(NO_3)_4$ sample is an insulator, bright regions are measured due to the charging effect during the recording of the SEM image, as shown in Figure 3. Although there are some cracks in the SEM image, its grain size is smaller than 1 μm.

3. Conclusions

In this work, we exhibited the results of XPS, Raman, and SEM for the $Th(NO_3)_4$ sample. In the XPS spectrum, we found the peaks involved in thorium, nitrogen, and oxygen atoms. Additionally, the Raman spectrum revealed the existence of thorium-oxygen bonding, anhydrous $Th(NO_3)_4$, complexed nitrate, and free nitrate. The morphology of $Th(NO_3)_4$ is also inspected by SEM experiment.

Fundamental chemical information on $Th(NO_3)_4$ will be utilized for further examination regarding the thorium material.

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