Resource Recovery from Advanced Ceramics by Hydrothermal Treatment

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INTRODUCTION

In order to realize the *materials' recycling society*, the development of the recycling and/or resource recovery processes with high efficiency and low energy consumption is required. However, the development of such process for the advanced ceramics is not enough. The hydrothermal treatment is one of the promising processes for recovering the valuable resources from various ceramics with the lower energy consumption. Moreover, the hydrothermal treatment is an '*earth-mimetic*' process, and thus is an '*eco-friendly*' process. In this paper, we demonstrate the advantage of this hydrothermal process for resource recovery of hazardous or precious chemical species (metals or anions in the solvent) from some advanced ceramics

EXPERIMENTAL

WC-Co cemented carbide, Nd-Fe-B sintered magnet and $LiCoO_2$ based-cathode were used as the treated samples. Each sample was sealed in the perfluoropolymer resin-lined pressure vessel with solvent, and then, was heated in an oven. HNO_3 and HF (for WC-Co), HCl and $(COOH)_2$ (for magnet) and water (for cathode) were used as solvents. After hydrothermal treatment for a given time, the precipitates were separated by the filtration, and were characterized by XRD. The filtrates were analyzed by the ICP-AES.

RESULTS & DISCUSSION

In the case of WC-Co specimens, tungsten trioxide hydrate ($WO_3 \cdot 0.33H_2O$) was collected by the hydrothermal treatment in the HNO_3, and the recovery yield and purity are ca. 99 mass% and more than 99.8 %, respectively, under optimal hydrothermal conditions (at 170^{o}C for 24 h in 7 mol/dm^3 HNO_3). Addition of HF remarkably decreased the treatment time. This acceleration is caused by the pitting corrosion of oxide layer coated on WC-Co specimen from HF. As a result from various investigations, the present *hydrothermal oxidation method* gave us a high purity $WO_3 \cdot 0.33H_2O$ powder regardless of the particle size of WC, Co content and the presence/absence of the corrosion resistant coatings.

The hydrothermal treatment of the Nd-Fe-B sintered magnet in the HCl and $(COOH)_2$ could precipitate Nd atoms as oxalate with 99.8 % of purity and more than 99 mass% of the recovery yield. The optimal conditions are at 110^{o}C for 6 h in the solvent with 3.0 mol/dm^3 of HCl and 0.2 mol/dm^3 of oxalic acid. Fe, Ni and B ions were remained in the treated solution. Although Fe and Ni ions can be removed as precipitates by pH adjustment, borate ions cannot be collected by the conventional method. We adopted a novel '*hydrothermal mineralization*' treatment method for wastewater polluted by borate ions. We succeeded in recovering borate ions dissolved in aqueous solution as calcium borate minerals by this process. The Nd oxalate and calcium borate would be supplied to the primary material production industry as resource.

As a result from the hydrothermal treatment of $LiCoO_2$ cathode, the Al-Co composite oxide was recovered as precipitates, and then, other elements (Li and F) were dissolved in aqueous solution. Here, Al and F atoms are originated from the electrical powder collector and polyvinylidine difluoride, respectively. Cobalt in the obtained Al-Co complex oxide could be recovered as metal by the conventional pyrometallugy.

Biographical Sketch

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Dr. Ryo SASAI was born in Hiroshima, Japan in 1969. He received his BS and MS degree in Materials Science from Hiroshima University in 1992 and 1994, respectively. His doctorate was earned at Hiroshima University in 1997 under the direction of Professor K. Gekko. Then, he moved to Graduate School of Engineering, Nagoya University as a postdoctoral fellow for JSPS Research Fellowship for Young Scientists. In 1999, he moved to Research Center for Advanced Waste and Emission Management (ResCWE), Nagoya University. He is presently an assistant professor of Division of Environmental Research, EcoTopia Science Institute, Nagoya University since 2004.

He received the Award from Tokai-Kagaku-Kogyo-Kai in 2003, and the Award from The Clay Science Society of Japan in 2006.

His research is focused on the following subjects:

(1) Development of resource recovery and recycling process with both lower energy consumption and higher efficiency of the various spent ceramics.

(2) Development of the novel materials or process for an effective decomposition of hazardous organic contaminants.

(3) Preparation of the organic/layered inorganic hybrid materials with highly luminescent property and its function-controlling by an external field.