Recovery of tantalum metal from used tantalum capacitor by using stream gasification

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Abstract

Recovery of tantalum from used tantalum capacitor is important because the price and the quantity of production of tantalum have not been stable, recently. However, recovery of tantalum from tantalum capacitor is difficult since the flame retardant and halogenated compound are contained in the tantalum capacitor. In this study we investigated by using steam gasification for recovery of tantalum, stabilization of halogenated compounds and hydrogen production. At first, we investigated the optimum conditions by using stream gasification with mixed molten carbonate for tantalum recovery rate. Secondly, we used sodium hydroxide (NaOH) by using steam gasification for the possibility tantalum recovery rate. Mold resin can be decomposed at low temperature, energy and short time by using stream gasification with NaOH.

Keywords: *Tantalum, metal, low temperature, stream gasification, sodium hydroxide*

1. Introduction

Tantalum metal is one of the rare metals and also the price and output are not stability. And it is called “conflict metal”, the argument involving the rights of mining has occurred in Congo which is one of the producing countries [1]. On the other hand, the high concentration tantalum is contained in the tantalum capacitor used for an electronic device. The development of recycling technology from used tantalum capacitors is required in order to reduce the environmental impact and the point of view of "urban mines". In the processing method, such as combustion and solubilization have been proposed previously [2], but this recycling methods are difficult because the chemical treating heavy metals or halogen compounds, thermosetting resin harmful. Therefore, the recycling of tantalum has not been carried out. In this study, we have investigated recovery of tantalum metal from used tantalum capacitor by using the steam gasification technology for the recovery of tantalum, stabilization of halogenated compounds and hydrogen production.

2. Materials and Methods

The tantalum capacitor (size: 6.0 × 3.2 × 2.5mm, model number: ESVC1V475M) was obtained from NEC /TOKIN Co.,Ltd. In this experiment, the catalyst was used molten carbonate (Na_2CO_3 : K_2CO_3 : Li_2CO_3)(1:1:1) and sodium hydroxide (NaOH). The experimental apparatus is shown in Fig. 1. The catalyst put into the reactor and heated by an electric furnace while flowing nitrogen (160ml/min) and steam (1ml/min) warmed by the heater. After heating to the reaction temperature of 530ºC, the tantalum capacitor (about 3g) was thrown in the reactor and the reaction time is 5~10 min.

Experiment (1); Setup of appropriate reaction conditions

Our previous study, separation of the tantalum of sintered compact was now possible by the steam gasification using mixed molten carbonate. Based on this result, we examined the suitable reaction temperature, the recovery rate of tantalum and the reaction time by using the mixed molten carbonate at 530 ºC for 5~10 min.

Experiment(2); Stream gasification by using NaOH

The experiment (2) was carried out based on the same conditions obtained in the experiment (1) and, using NaOH at 530 ºC for 5 to 10 min. Sodium hydroxide can be expected more hydrogen production and lower cost than the molten carbonate.

Experiment(3); Cause gasification occurs at low temperature

We investigated the reason of gasification occurs from about 500 ºC by using mold resin and change the amount of NaOH and nickel.In the experiment (3) was gasified to 650 ºC under the following conditions,

①mold resin (1.3g)
② mold resin +nickel (1.3g+0.1g)
③ mold resin + NaOH (1.3g+1g)
④ mold resin + nickel + NaOH (1.3g+0.1g+1g).

After the experiment, the product gas was analyzed by a gas chromatograph (GC-2014ATF) made by SHIMADZU. The residue remaining in the reactor was allowed to melt deposits on a hot stirrer charged with distilled water and stir. The water that used bubbling and stirred was analyzed by ion chromatography (DX-120).

3. Results and Discussion

Figure 2(a) and 2(b) are shown the sintering compact after the experiment by using the mixed molten carbonate at 530 ºC for 5~10 min. In the case of 10 min, the sintering was broken while the reaction time 5 min, the tantalum was not broken and, able collect from all the samples. This is considered that if the reaction time was

long, the carbon contained in the sintered compact is also gasified.

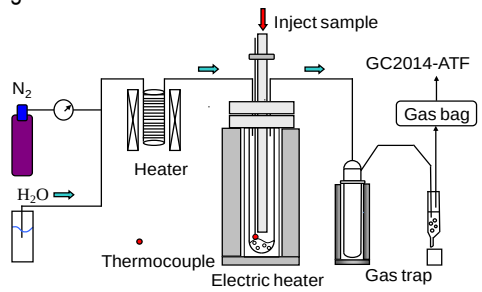

Fig.1 Experiment apparatus of stream gasification

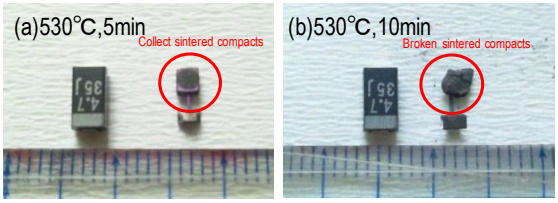

Fig.2 Effect of reaction time at 530ºC

In the second experiment was carried out when only steam gasification, the molded resin was left and could not complete separation (Fig. 3 (a)).If sodium hydroxide was also used,the mold resin is broken, and sintered compact is exposed (Fig. 3 (b)). Figure 4 shows a comparison of the effect on the product gas of steam, carbonate, and sodium hydroxide.From these result, we understand that the amount of hydrogen production was significantly increases when using NaOH. The CO and CO_2 gas are produced OCN epoxy resin which have a main component of the organic matter in the molding resin and OCN epoxy can react by the water gas shift reaction . Table 1 show the analysis of the halogen concentration of residue. From the results we understand that sodium hydroxide can react on the remaining residue and was able dehalogenation.

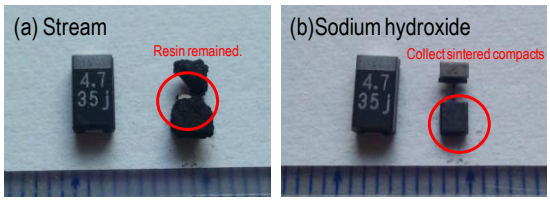

Fig.3 The sample collected after the experiment (530ºC, reaction time 5 minutes)

Table 2 shows the ratio of the mold resin composition before and after the experiment.Since the carbon of mold resin before the experiment was about 16%, it was reduced to 10% after experiments.Therefore, we were able to confirm the gasification reaction at 530ºC. Figure 5 shows the hydrogen produced in experiment 3. Since the amount of hydrogen is not increased under 600ºC by using only stream gasification, we considered that the active gasification does not occur 500ºC. When using Ni, the amount of hydrogen is greatly increased compared with only water vapor from 550ºC. The gasification of hydrogen producted was occur from 550ºC when using NaOH.But the addition of Ni and NaOH could be confirmed that the production of active hydrogen from 500ºC.We suggested that gasification happened from 500 ºC in the gasification of tantalum capacitor, NaOH and nickel in the tantalum capacitor terminal are reacted as a catalyst.

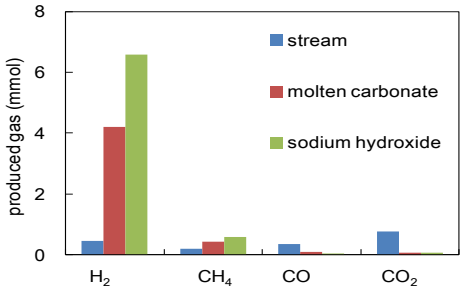

Fig.4 Effect on the product gas of steam, carbonate, and sodium hydroxide (530ºC, reaction time 5 minutes)

Table.1 Halogen concentration of residual substance

Halogen concentration of residue (ppm)

F	Cl
43.42	16.81

Table.2 Composition of resin before and after the experiment

The result of ultimate analysis (wt%)

	C	H	N
Before	15.94	1.59	0.12
After	10.38	1.37	0.00

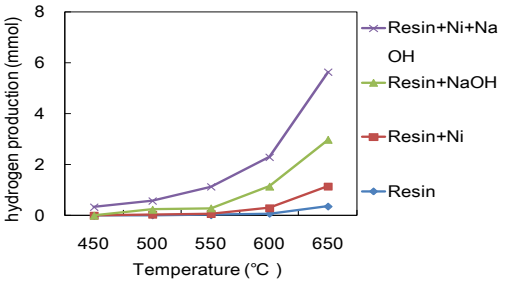

Fig.5 The amount of hydrogen generation in gasification of mold resin

4. Conclusions

The tantalum sintering compacts could collect from all the samples by using NaOH and molten carbonate at 530 ºC for 5 min. The stream gasification was occured at low temperature because the terminal of Ni in the capacitor and NaOH were reacted. Therefore, we can expect, the tantalum metal can separate and collect by using stream gasification with NaOH.

Reference

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[2] Tohru Kamo, Scientific research for promoting formation of a recycling society, p.4 (2010)